VACUUM

A Review of Developments
in
Vacuum Research and Engineering

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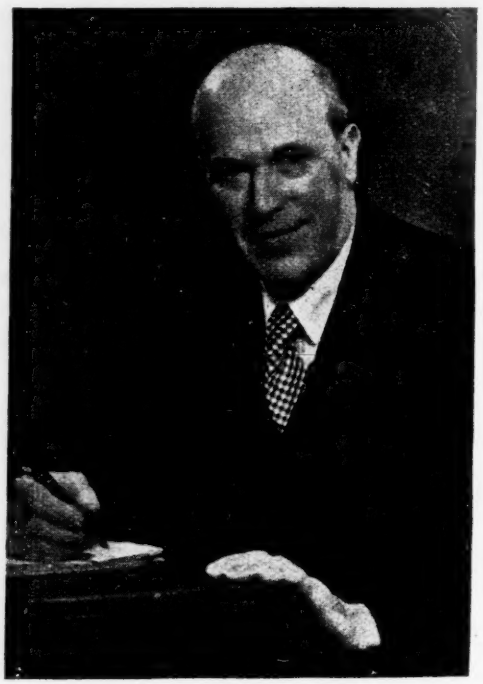
1
951

FOREWORD

ALTHOUGH THE USE OF VACUUM is now so important to Science and Industry and references to its use are constantly found throughout scientific and technical literature devoted to fields as diverse as Engineering and Pathology, no journal in the English language adequately reports progress in this vital subject, so that much of importance escapes high vacuum workers. ' VACUUM ' is published in the hope of making up this deficiency and thus lightening the task of all who wish to keep abreast of modern developments.

Authoritative articles by vacuum specialists in all parts of the world will keep readers in touch with the latest vacuum procedures employed with success in various arts and sciences, and in many cases it will be found that similar methods will find application in other and diverse fields. It is hoped that our correspondence columns will be both interesting and informative.

An essential feature of this journal will be the comprehensive abstracts of vacuum work compiled from world publications by our highly specialised abstracting organisation and the efficiently detailed classification index will facilitate easy reference and permanent recording.



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Formerly Quain Professor of Physics in the University of London, where he established an important school of research on the fundamental mechanical properties of the solid and liquid state, distinguished by the elegance of its experimental technique. Vacuum work has played a large part in his researches, an interest he has maintained since his Heidelberg days under Lenard, and has found many applications, for instance, in his study of the liquid metal viscosities and the preparation of single crystals of metals. He wrote the article on Vacuum in the current edition of the Encyclopaedia Britannica and recently delivered a Friday Evening Discourse at the Royal Institution on High Vacuum. A basic law concerning the viscosity of liquids and an important equation expressing the creep of metals bear his name. He has exerted a powerful influence on a wide variety of scientfic problems. He has an exceptional literary talent which is evident in his scientific writing and has brought him renown as a creative poet. He is also one of the great lecturers of our time. His extensive knowledge of the history of science is widely recognized ; he is a leading authority on Isaac Newton and recently delivered the Wilkins Lecture of the Royal Society on Robert Hooke. He is a noted bibliophile and possesses a remarkable collection of early scientific books.

Professor Andrade was born in London in 1887 and graduated at London in 1907, after which he carried out research at the Universities of London, Heidelberg, Cambridge and Manchester. His honours are numerous : he is a Doctor of Philosophy of Heidelberg, Doctor of Science London, Hon. LL.D. of Edinburgh, a Fellow of the Royal Society and its Honorary Librarian, a Chevalier of the Légion d'Honneur, and also possesses the jealously guarded distinction of Correspondant de l'Académie des Sciences de l'Institut de France.

THE SERVICES OF VACUUM

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Summary

THE AUTHOR SURVEYS the field of Vacuum Techniques from their inception as a laboratory practice, and subsequent improvements of basic apparatus leading to the industrial applications of to-day. The causes are studied which led to the rapid developments of the last few years and established vacuum processing as a vital requirement for research and industry.

Sommaire

L'AUTEUR FAIT UN EXPOSE du vide sous ses divers aspects : l'origine de son usage dans les laboratoires, suivi par l'amélioration des appareils aboutissant aux applications industrielles d'aujourd'hui. Il cherche ensuite les causes du développement rapide de ces dernières années qui a rendu la technique du vide indispensable dans les laboratoires de recherche ainsi que dans l'industrie.

Vol. 1 1951

ENGINEERING, and industry in general, have for centuries made use of scientific discovery and built machines, erected structures, devised processes and produced materials which owe their success to the just application of general scientific principles and particular observations. The last thirty years or so, however, have seen a new phenomenon, the direct transference to the factory and the industrial plant of inventions, techniques and processes born in the academic laboratory, without other modifications than those necessary for more economical and speedy functioning on a larger scale. An example that readily occurs to everyone is the modern bulk manufacture of thermionic valves and vacuum tubes in general, and of devices embodying them, sometimes called the light electrical industry to distinguish it from the long established industry that deals with heavy machinery and with power transmission. The cathode ray tubes of industry are much like those developed for use in the laboratory; the valves are versions, very much improved if you like, of those born directly from academic physics. The chemical industry offers another example of laboratory methods and techniques adapted with little alteration to large scale production; the manufacture of modern drugs and biological preparations is academic organic chemistry enlarged. Today we are, as a result of recent developments, confronted with yet another example of a tool of the research laboratory becoming an engineering triumph and industrial adjunct, namely the intensive development of large apparatus for rapidly producing really high vacua, which has become an essential factor in a range of very diverse processes. The production of the modern vacuum pump and its accessories is at the present time a highly specialised, ingeniously developed and rigorously controlled branch of precision engineering in which England can confidently claim to be well in the lead.

The automatic high-vacuum pump of the physics laboratory was born within the experience of men still in full vigour. In practically all the classical work of the Cavendish laboratory under J. J. Thomson the vacuum was produced with the old Töpler form of mercury pump, which demanded hours of patience and, in the final stages, some skill, to produce a cathode ray vacuum. Forty years ago it was the ambition of the English research worker to have the use of a Gaede rotating mercury pump, in which segments of an ingeniously designed drum, turning in an enclosure containing mercury, were alternately filled with gas from the vessel to be exhausted and with mercury: the principle was

* M.S. Received September, 1950.

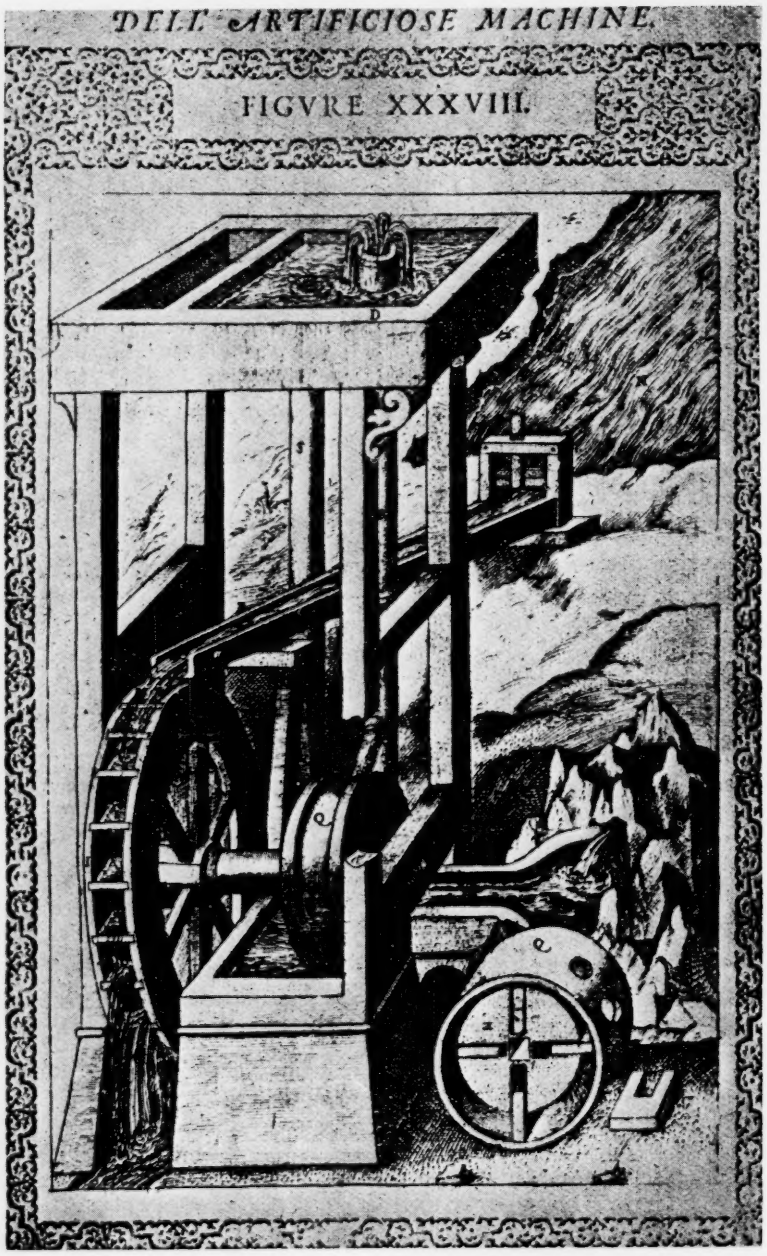

Fig. 1.—Ramelli's Rotary Pump with vanes. An outside view of the pump, which is driven by a water wheel, appears a little below the centre of the picture: the construction of the pump is shown in the lower right hand corner of the picture.

Vol.
1
1951

similar to that of the commercial gas meter, only that in the meter the gas effects the rotation, while in the pump the rotation expels the gas. This was the first efficient pump of modern type, in that it would run continuously without attention and produce a vacuum of 0.00001 mm. of mercury.* Like most modern pumps for high vacua it required a backing pump: it could not function until a rough vacuum had already been produced. Just before the first world war Gaede devised a pump on a completely new principle, the so-called molecular air pump, which depended for its action on the fact that a moving surface communicates a component of velocity in the direction of its motion to gas molecules which strike it, so that if the pressure is already low—the mean free path large—the gas can be dragged along. The surface speed of the rotating cylinder which forms the heart of the pump must be very high, the construction very accurate and a good backing pump is necessary. Holweck produced another form of pump acting on this principle and Siegbahn still another form. Molecular pumps have, among other advantages, that of dealing equally well with condensible vapours and with gases, but on account of mechanical difficulties of construction they were never extensively produced and, while they are regularly used in a few laboratories, their adoption has been generally hindered by the great development of the simply made diffusion pump.

One of the most widely used pumps today is the mechanical rotary pump, which, in principle, is nearly four hundred years old, as can be seen from the diagram, taken from Ramelli's book of 1588, shown in Fig. (1). An improved type is Prince Rupert's "water-bolt", made about a hundred years later, see Fig. (2). These pumps were, of course, from their rough construction, suitable for liquids only. It was Gaede again who first made a practicable metal air pump of this type, producing a vacuum of about 0.01 mm. mercury, see Fig. (3). Such pumps are now manufactured on a large scale in a great variety of sizes, and, owing to improved design and special oil, produce vacua of 10^{-3} mm. absolute and partial pressures of air of about 10^{-5} mm. mercury,

*The figure claimed, which I do not guarantee.

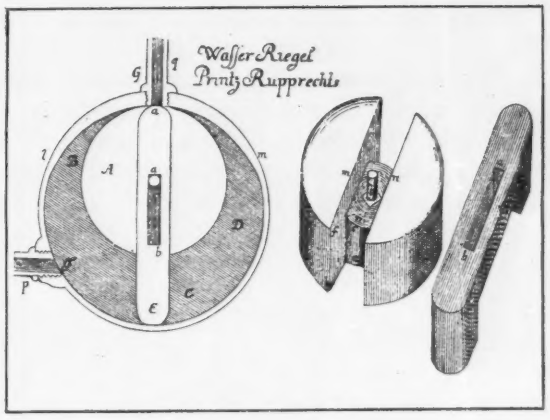

Fig. 2.—Early Rotary Pump.
Prince Rupert's "Water-Bolt", designed in the middle of the seventeenth century as a water pump. The picture is from Leupold, *Theatrum Machinarum Hydraulicarum*, 1724. (Compare with the design of that type of modern pump called the "Kapselpumpe", by Gaede.)

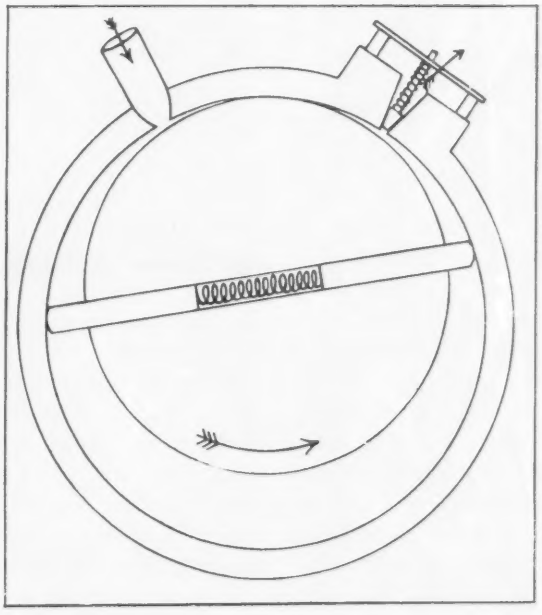

Fig. 3.—Modern Rotary Air Pump.
Gaede's *Kapselpumpe* (Compare with *Fig.* 2.)

the difference being due to the vapour pressure of the oil.

The backing pumps needed by modern pumps which produce the highest vacua are nearly all of this type. The smallest laboratory types have a speed of about 10 litres per minute and the largest types in general use a speed of about 3,000 litres per minute.

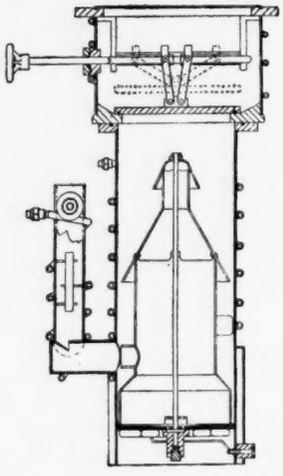

Fig. 4.—A typical oil diffusion pump, having three stages. The bore is 9 inches.

The high vacuum pump most widely used today, the diffusion pump, is again a product of Gaede's genius: he first put forward the principle in 1915 and soon after the close of the first world war many ingenious variants of his design were current, both in glass and metal. The principle is that a swiftly moving, approximately unidirectional, stream of vapour is produced, into which the molecules of the gas to be removed diffuse and are carried away. All types of diffusion pumps need to be used in conjunction with a so-called backing pump capable of producing a pressure varying, according to the type, from 0.01 to a few mm. mercury, the exact value depending on the type. The liquid originally used to produce the vapour stream was mercury but today the low vapour-pressure oils of the type first produced by Burch are widely used and the liquid butyl phthalate and certain of the silicones also have advantages. All technical diffusion pumps in use today have two or more stages, acting successively (Fig. 4). In general the design of the pump, the choice of the liquid and the selection of the most efficient size of backing pump are highly technical matters, involving the kinetic theory of low pressure gases, as first developed by Knudsen, the thermal stability of the liquid and mechanical and thermal considerations. The pipe lines, the taps and the valves concerned also demand expert knowledge. The days when one just ordered a large vacuum pump are as remote as the days when one just bought an electric motor. Every technical vacuum problem today is a matter for experts.

The measurement of low pressures is also a matter that has developed into a branch of applied science. The old McLeod gauge still has its uses, and mechanical gauges of both the Bourdon and the diaphragm types are used for non-corrosive gases down to pressures of a tenth of a mm. mercury or so, but for the measurement of very low pressure the special thermal, mechanical and electrical properties of very tenuous gases are all effectively employed. The viscosity is used in the Dushman gauge: the radiometer action, which is a thermal effect shown by low pressure gases, in the various forms of Knudsen gauge: the thermal conductivity of the gas in the Pirani gauge: the electrical conductivity in the many forms of the ionisation gauge. All these properties vary, of course, with pressure when the pressure is low. In one of the latest forms of ionisation gauge a small quantity of radium produces the ionisation, which is measured by the saturation current, a suitable amplifier being used. The Pirani and the ionisation gauge are the types most frequently employed in large installations. These problems of pressure measurement are particularly good examples of the way in which delicate problems of academic physics find application in modern vacuum work. Knudsen's classical researches on the theory of the behaviour of gases at low pressure find further application when the

Vol. 1 1951

design of vacuum pipe lines is in question.

Before we turn to the various applications of the high vacuum, let us see in a general way what the effect of removing air (or, for that matter, any other gas) will be on physical phenomena. Encounter with gaseous molecules hinders the passage of all atomic and sub-atomic particles—electrons, alpha particles, metallic atoms, the molecules of the vapour leaving a liquid surface. Hence, supposing that we want to accelerate electrons or gaseous ions in an electric field, we must remove the air from the path as much as possible: the higher the vacuum, the fewer the encounters. All electronic and atomic accelerating apparatus, therefore, from the valve and the cathode ray tube in the television set to the largest betatron and cyclotron, from the electron microscope to the largest mercury rectifier, require suitable vacuum equipment in their manufacture or for their operation. Or, again, supposing we want a free run for the atoms leaving a hot metal or the molecules leaving a liquid surface, either to obtain a deposit in the former case or to vaporise the liquid at low temperature in the latter, a high vacuum is required. For free distillation at low temperature it is necessary to give the molecules leaving the liquid surface an unimpeded run: the removal of gas from molten metals likewise demands a removal of any atmosphere, the impact of whose molecules would drive back the gas molecules striving to escape. The removal of atomic and molecular obstacles is, then, the general end of vacuum technology.

Let us now look at some of the chief uses of modern high vacuum equipment. Every physical and chemical laboratory today, whether academic or industrial, contains a few small vacuum outfits, usually a diffusion pump with its backing pump, for routine use, quite apart from more elaborate installations for special researches. All X-ray outfits, except those making use of sealed-off tubes; mass spectrographs; vacuum spectrographs for soft X-rays; vacuum stills, to name the first instances that come to mind, require good vacuum equipment, but a dozen other uses will readily occur to the reader. Much larger outfits are required for the various types of particle accelerators, such as the cyclotrons, the synchrotrons and the linear accelerators, since here the charged atoms, such as deuterons, have to travel in some cases many miles, or tens of miles, during acceleration. Thus we are told of the three thousand million electron-volt accelerator now being planned at the Brookhaven National Laboratory, New York, that the maximum permissible pressure in the high vacuum chamber is 10^{-5} mm. of mercury, which will demand twelve or more pumping stations each consisting of a 20 inch oil diffusion pump, suitably backed; the speed at the entrances will be about 2,500 litres per second for each pump. So far have we travelled from Cockcroft and Walton's original atomic accelerator, which was satisfactorily exhausted by one oil diffusion pump of 300 litres per second. Using what is an essentially laboratory method we have passed into the vacuum engineering stage.

When we consider the great atomic establishments, such as Harwell and the larger concerns in the U.S.A., enormous high vacuum installations are essential, although of them, of course, little has been published. We have Dushman's authority for the existence in 1947 of oil diffusion pumps with a performance of above 7,000 litres per second. As he says " The manner in which these high speed pumps contributed towards the success of the Manhattan project and other developments during the recent war is too well known to require more than mere mention." In fact, it is not too much to say—no modern vacuum pumps, no atomic bomb.

In these atomic matters we are concerned with particles flying very large distances at, in the later part of their travel, very high speeds, when the scattering at a single encounter is relatively small. When we come to consider atoms and molecules moving with thermal speeds the distances travelled are relatively small but an encounter is a serious matter, so that once more very high vacua are necessary. One field in which such particles are concerned is that of vacuum coating. If a metal be heated in a high vacuum the atoms evaporating from the surface fly in straight lines and deposit on any clean surface suitably placed; with many metals the

vacuum is also necessary to prevent oxidation. This evaporation technique is extensively used for the preparation of front-coated mirrors on glass: for the so-called "blooming" of lenses and mirrors (which consists in the deposition of a film of, for instance, magnesium fluoride, of a thickness chosen so as to cut down reflection) and for the preparation of the new high reflection film used for coloured filters: for the deposition of metal films on plastics for scientific and for decorative purposes: and for many other purposes where the lack of heating of the surface and the ease of control of thickness are advantageous. A very interesting development is the deposition of zinc and aluminium onto paper strip for use in condensers. The 'shadowing' of specimens for examination in the electron microscope with a heavy metal is another example of the use of the vacuum evaporation technique. The method of cathodic sputtering is also used for depositing a coating of certain metals, but for this a much less perfect vacuum is necessary, to carry the discharge.

The evaporation of liquids in high vacua has in the last few years found a range of applications that have led to heavy demands on the vacuum industry. High vacuum distillation is the oldest application, having been used by Burch more than twenty years ago for the preparation of his famous "apiezon" low vapour pressure greases and oils, which have rendered such signal service in high vacuum technique: as he then pointed out, when the molecules of the distillate vapour collide more frequently with the walls of the still than with the residual gas molecules, the latter have substantially no influence. The temperature of distillation is, of course, reduced far below that necessary when atmospheric pressure prevails, which means that substances prone to thermal decomposition can be safely handled. To-day high vacuum distillation is used for a great variety of purposes in the chemical industry and at least six different types of stills are available. The method is used for such purpose as the separation and concentration of components of animal fats and oils, and of organic compounds of high molecular weight in general, such as sterols, vitamins and free fatty acids. The separation of vitamin A from cod liver oil and of sitosterol from vegetable oil may be cited as particular examples.

The fact that free vaporisation takes place at a comparatively low temperature when the prevailing pressure is very small is utilised today for the rapid drying and concentration of heat-sensitive materials. Orange juice may be taken as an example, the object being to produce a powder, or liquid concentrate, which on the addition of water shall give a product similar to the fresh juice. It is said that this is attained if the fresh juice, or juice somewhat concentrated, is rapidly evaporated at about 50°C. under a pressure of a few mm. of mercury. The same process has apparently been applied to coffee, milk and other food products. More important, however, is the drying of various important biological materials, such as blood plasma and serum. Here drying is essential where storage is contemplated not only on account of bulk but because changes take place in the stored liquid even at temperatures round about freezing point. The technique adopted is that termed 'freeze-drying'. It is well known that at very low pressure, ice rapidly sublimes—passes from the solid to the vapour state—the vapour pressure being 4.6 mm. of mercury or below according to temperature. The material is, therefore, rapidly frozen by special methods and exposed to very low pressure, the water vapour that is given off from the frozen substance under these conditions being condensed at low temperature, absorbed by desiccants or directly removed by pumping. With freeze-drying there is no foaming such as is liable to take place with a liquid; there is, owing to the low temperature, and to the absence of free liquid, ordinarily no chemical change or bacterial growth during the drying; and the product is left in a porous sponge-like state which makes rapid re-constitution easy. Further, owing to the low pressure, oxidation is restricted. The process is applied today on a large scale not only to human blood serum and plasma but to penicillin and other antibiotics, to antitoxins, to certain proteins, to vaccines, and to the preservation of living biological material, such as bacterial cultures, virus suspensions and various tissues.

Another example of the large scale use of high

Vol. 1 1951

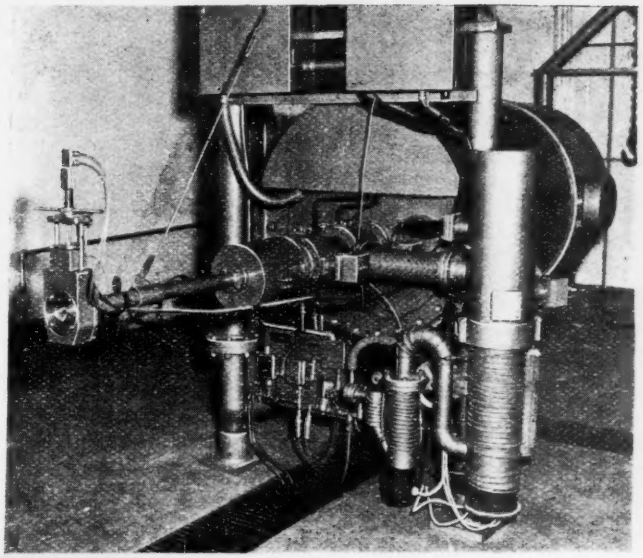

Fig. 5.—Two-Million-Volt Electron Accelerator for Radiation Research. The vacuum system is located to the right with the mechanical pump in the background. This generator is a source of high-energy rays or x-rays (see page 7).

by courtesy of High Voltage Engineering Corp., Cambridge, Mass., U.S.A.

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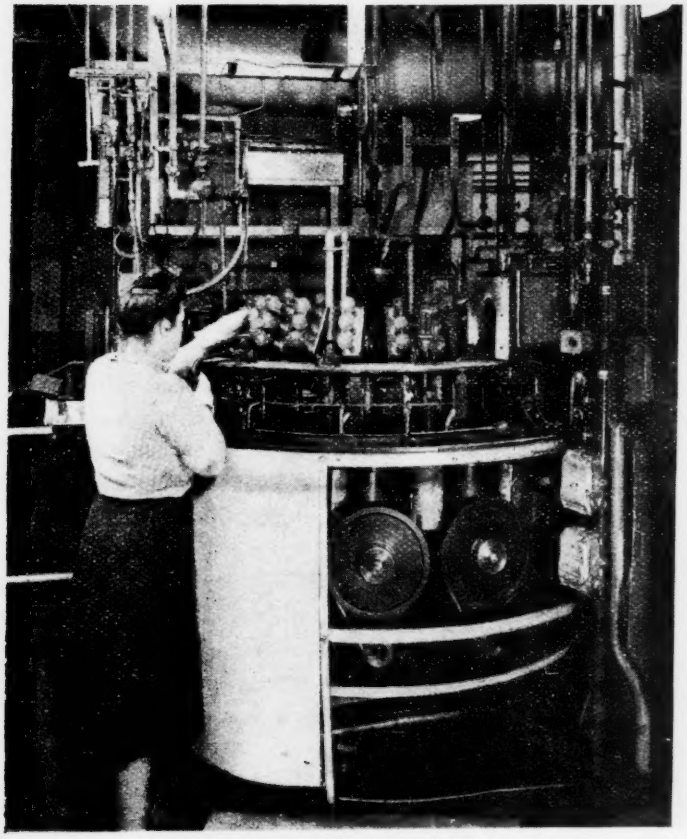
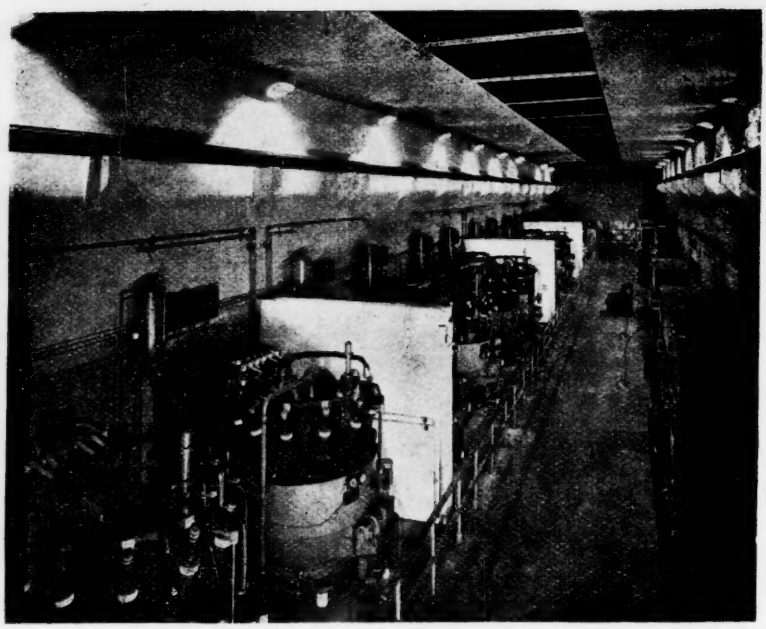


Fig. 6.—Exhausting of Rectifier Valves. An automatic 16-head pumping plant with interchangeable individual units consisting of a rotary and a diffusion pump. Output of the machine: 75 valves per hour. (see page 10).

by courtesy of M. O. Valve Co. Ltd., London, England.



by courtesy of The English Electric Co., Ltd., London, England.

Fig. 7.—An installation of water-cooled, continuously exhausted steel-tank type mercury-arc rectifiers with a total output of 41,200 amperes at 675 volts, for the electrolytic production of aluminium. Each rectifier unit is served by a rotary and diffusion pump permanently exhausting the rectifier at a pressure of the order of 0.001 mm.Hg. The Pirani gauge operates a safety relay in the event of an excessive pressure rise in the system.

Vol.
1
1951

vacua occurs in metallurgy, where today charges weighing hundreds of pounds are melted and cast under low pressure conditions. Two obvious advantages are that the molten metals are protected from attack by gases and that any dissolved gas is discharged. Further, in certain cases reactions which are desired, such as that between a metallic compound and a reducing agent, are favoured by low pressure. In general, vacuum methods of refining metals, which twenty years ago were mainly carried out by Heraeus, are coming more and more into favour. The pressures vary from 1 to 0.005 mm. of mercury, according to the object in view.

Nothing has been said of the use of vacuum plant for the production of electric lamps, valves, cathode ray oscillographs and suchlike, since this older technique, with its use of getters which combine with and fix the residual gas, is familiar, but it remains one of the most essential applications of the art of exhaustion as far as the general public is concerned. Nothing has been said of the indispensable craft of making suitable taps, valves and joints, or of detecting leaks. Every reader has, no doubt, experience in some part of the vacuum field; the desire has been to put before him briefly some indication of the surprising scope of the subject today. And, if the writer, who has watched the progress of Mr. F. D. Edwards' undertaking since its small, its very small beginning, may end on a personal note, there is also a desire to express to the energetic and expert company whose latest enterprise this journal represents the warmest good wishes for the success of the venture. It may be predicted with confidence that very little unwanted gas will ever be found to have leaked into VACUUM.

A REVIEW OF LABORATORY FREEZE-DRYING with particular reference to viruses

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Summary

THE following aspects of freeze-drying are briefly considered, firstly, the physical principles; secondly, the objectives of this method of desiccation; thirdly, application of the method to: (1) the stabilisation of enzymes, (2) the preservation of body fluids and of media for bacterial and tissue cultivation, (3) the preservation of bacteria, (4) the preservation of plant and animal viruses, and (5) the preservation of tumour-producing viruses with special reference to the claims of Gye and Craigie, and to the work of the author and his collaborators on the preservation of fowl tumour viruses. Lastly, a few of the technical difficulties, and problems, are considered which remain to be solved.

Sommaire

ON considère trois aspects de la dessication à froid sous vide, premièrement les principes physiques, secondement les buts de cette méthode de séchage, troisièmement l'application de la méthode à: (1) la stabilisation des enzymes, (2) la conservation des liquides corporels et des milieux pour la culture des tissus et des bactéries, (3) la conservation des bactéries, (4) la conservation des virus d'origine animale ou végétale, (5) la conservation des virus produisant des tumeurs avec référence spéciale aux revendications de M. M. Gye et Craigie et au travail de l'auteur et ses collaborateurs concernant la conservation des virus produisant des tumeurs chez les poules. Finalement, on considère certaines difficultés techniques et des problèmes restant à résoudre.

INTRODUCTION

THE PROBLEM of the storage of labile biological materials, such as proteins, enzymes, bacteria and viruses, is one of considerable importance and complexity. Separated from the living organism proteins tend to undergo an irreversible process known as " denaturation ". Denaturation is a result of the aggregation of the protein molecules, and the denatured protein is usually insoluble and devoid of the specific properties—*e.g.*, enzymic or antigenic—of the native protein.

Thermal and surface forces, in particular, are responsible for denaturation; therefore, for optimum preservation, storage procedures must be devised in which these forces are minimal. In general, storage at low temperature favours the preservation in a native state of a protein in solution. A marked improvement is obtained, if the storage temperature is kept at a figure below the freezing and partial eutectic point of the solution used. Below the temperature where the whole mass becomes solid preservation is excellent. For serum the eutectic point (−21.4 °C) is that of a sodium chloride solution. There will be no liquid phase below this temperature and the serum proteins will be virtually " dry ". In the absence of a liquid phase bacteria do not multiply and therefore the contamination of the dry protein by bacterial growth during storage is kept to a minimum.

Prolonged low-temperature storage of biological materials in the condition just described is an uneconomic procedure calling for large-size refrigerators and involving difficulties in transportation. If, on the other hand, the water content of the preparation is considerably reduced and maintained at a low value, the material is diminished in mass, and, in addition, may be stored for long periods at room temperature.

The removal of water from a solution requires the use of heat energy and, since a rise of temperature usually accelerates denaturation, the problem of

* MS. Received July 1950.

drying is one of putting in the necessary heat with the least possible destruction of material. A variety of such methods exist and a number of them were reviewed by Greaves in 1946 [23]. Chemical methods of water removal involving low-temperature precipitation with alcohol or alcohol-ether mixtures are often unsuitable for lipoid-containing materials where the lipoids are an essential component [6 7 8]. High-temperature methods such as the roller- and film-drying techniques are only of limited application, *e.g.*, the production of dried milk. Spray-drying is the least destructive of such methods, and has been used—on a restricted scale only—for the drying of human serum and plasma.

The removal of water by distillation or evaporation lends itself to the drying of biological materials, provided that the temperature required for the treatment is not so high as to endanger the composition of the material. It is obvious from the foregoing statements that drying from the frozen state ideally fulfills these requirements. If, in order to accelerate drying, the partial pressure of air surrounding the frozen material is reduced to a value lower than the partial pressure of vapour in contact with it, sublimation of ice occurs. Heat may then be supplied at a low temperature to maintain sublimation without causing an increase in the temperature of the material. Sublimation thus carried out under vacuum constitutes the basic principle of the technique known as ' freeze-drying '. It is the method of choice for drying heat-labile proteins, enzymes and viruses.

GLOSSARY OF TERMS

Acacia. Gum acacia (gum arabic) occurring in nature.
Aetiology. The study or theory of the causation of disease.
Antigen. Any substance which, introduced into a living body, incites the formation of antibody.
Bacteriophage. A virus which produces a transmissible effect on bacteria associated generally with lysis of the bodies of the organisms.
Bovine Testis Hyaluronidase. An enzyme, produced by the bull testis, which hydrolysis hyaluronic acid.
Carcinoma. A malignant tumour arising from epithelial tissue.
Catalase. An enzyme which decomposes hydrogen peroxide.
Ceralose. A form of commercial glucose.
Cysteine. An amino-acid containing sulphur.
Enzyme. A substance, usually a protein, produced by living cells and capable of producing the transformation of some other substances by catalytic action.
H. Pertussis. The causal agent of whooping cough.
Herpes Simplex. An inflammatory skin disease characterised by the formation of small clusters of vesicles.
Hog Cholera. An acute infectious communicable disease of swine caused by a virus.
Hydrolysate. The end product of hydrolysis.
Isotonic Solutions. Solutions having the same osmotic pressure as body fluids.
Lipoid. Resembling fat.
Lymphocytic. Pertaining to lymphocytes—a type of white blood corpuscle which arises in the lymph glands.
Lymphogranuloma. A disease characterised by the progressive enlargement of the lymph glands.
Lyophile. Capable of being readily dispersed in solution.
Lysates. Material formed by the lysis of cells.
M. Myco-bacterium.
Mucin. A group of glyco-proteins contained in the secretion of certain glands.
Myosin. A globin, which is the most abundant protein in muscle.
Papilloma. A non-malignant wart.
Peptone. A product obtained by the progressive action of enzymes or acids on proteins.
Phosphatase. An enzyme which transforms phosphorus compounds.
Pneumococcus. The organism, Diplococcus pneumoniae, which causes lobar pneumonia.
Polypeptide. A compound formed by the union of more than three amino acids.
Protein. Nitrogenous compounds, combinations of α-amino acids and their derivatives, forming the characteristic constitutents of the tissues and fluids of the animal body.
Reticulum. A fibrous network.
Salmonella. Gram-negative, non-spore forming, primarily intestinal, bacterial parasites.
Sarcoma. A malignant tumour of connective tissue origin.
Serratia Marcescens. Bacillus prodigiosus, one of the smallest of bacteria producing a bright red pigment.
Steroids. A group name for compounds related chemically to cholesterol, such as sex hormones, bile acids.
Thiourea. Thiocarbamide, $NH_2.CS.NH_2$.
Titre. The measure of the quantity of a substance required to produce a given reaction.
Treponema Pallidum. The causal agent of syphilis.
Tyrode's Solution. A solution containing 0.8% sodium chloride, 0.02% potassium chloride, 0.02% calcium chloride, 0.01% magnesium chloride, 0.005% monosodium phosphate, 0.1% sodium bicarbonate and 0.1% glucose.
Viability. Ability to live.
Virus. Disease-producing organisms, much smaller than bacteria, and unlike these, able to reproduce only in association with living cells.

Vol. 1 1951

RESUME OF THE TECHNIQUE

The desiccation of proteins by high-vacuum sublimation from the frozen state was first used by Shackell in 1909 [26] [47]. The protein solutions froze as a result of rapid evaporation of the water, which was removed from the system by absorption in sulphuric acid. For industrial operation the chemical desiccant was replaced by cold condensers [15]. Frothing, which is an undesirable characteristic of the evaporation method, was originally overcome by pre-freezing. More recently, however, two alternative procedures have been used; degassing of the protein solution prior to snap freezing by evaporation [24a] and suppression of frothing by centrifuging the material during evacuation [22].

The material is maintained in the frozen state due to the high latent heat of ice and the high rate of sublimation obtainable under vacuum. If efficient water vapour-collecting means are provided, the rate of drying depends only upon the rate at which heat is supplied to the frozen mass.

If pre-frozen material is to be dried the pressure in the vacuum system must be reduced as rapidly as possible to a point low enough to facilitate rapid sublimation; the frozen material will otherwise partially or completely melt. In practice, freeze-drying is carried out in the pressure range, 3 mm. mercury (=3,000 microns) to 0.01 mm. mercury (10 microns).

The moisture content of the dried material depends solely upon the equilibrium vapour pressure conditions existing in the drying apparatus. 0.5% by weight of the dried solids is a typical water content claimed for many freeze-dried products.

OBJECTIVES OF THE METHOD

The objectives in freeze-drying have been very aptly described in E. W. Flosdorf's latest publication [20] as follows :—

(1) Production of Minimal Chemical Change

Drying at low temperatures avoids much of the chemical change that accompanies evaporation at normal temperatures. Many viruses may be held in an active state under frozen storage conditions, but other materials such as guinea pig complement are difficult to store successfully even in a refrigerator. Between these two extremes it is true to state that freeze-drying is the only method of drying such materials under conditions in which chemical changes are minimal.

(2) Prevention of Foaming

The majority of proteins are readily denatured by surface spreading at interfaces and, for this reason, excessive foaming must be avoided during drying. Pre-freezing, or centrifugal vacuum freezing (introduced by Greaves in 1944) [22] entirely obviate this difficulty.

(3) Decrease in Aggregation

The products of the freeze-drying of proteins are usually highly-porous reticula which readily redisperse on addition of water. Gelatin, for example, which is normally readily dissolved only in boiling water, becomes easily soluble in cold water after freeze-drying.

(4) Sterility

In the modern freeze-drier it is a simple matter to ensure the sterility of the product during drying and, similarly, the final dry product is resistant during storage to enzymic action and bacterial growth.

(5) Minimal Oxidation

The dry product may either be stored *in vacuo* or the storage vessel (bottle or ampoule) may be filled with a dry, oxygen-free, inert gas such as nitrogen.

(6) Ease of Storage

It is self-evident that a dry solid is easier to store and transport than a solution or suspension. The particular dry material may require to be stored at refrigerator temperatures but, in general, proteins are more resistant to heat denaturation when they are dry than when they are in solution.

(7) Prevention of Biological Change

Freeze-drying enables bacteriologists to preserve original cultures of live bacteria, and to avoid the

degenerative changes which occur when an organism has to be maintained by frequent sub-culture [43].

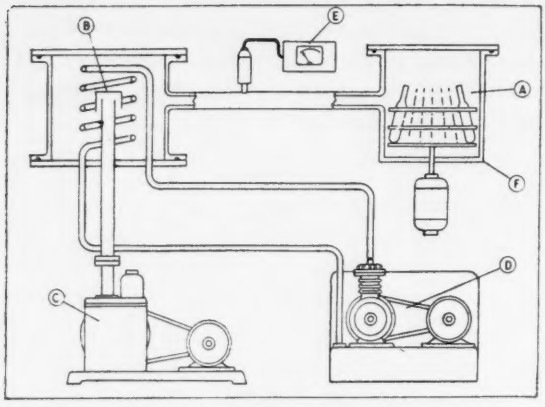

Fig. 1.—Schematic arrangement of Centrifugal Freeze-Dryer. (A) Drying Chamber; (B) Refrigerated Water Vapour Condenser; (C) Vacuum Pump; (D) Refrigerator Unit; (E) Vacuum Gauge; (F) Centrifuge Assembly.

APPLICATIONS

The greatest single stimulus to freeze-drying has undoubtedly been the need for blood serum and plasma in World War II to meet civilian and military requirements. Greaves' plant in Cambridge [23] alone produced dried blood products corresponding to more than 10,000 litres of blood between June, 1940 and September, 1945. Much of the recent research work based on the freeze-drying technique has been carried out on apparatus fabricated *ad hoc* by the various research groups. No doubt many satisfactory "home-made" freeze-driers exist, [5 21 27 51 61] but there are many advantages in the use of a commercial apparatus which will give consistently reproducible results.

Freeze-drying has been applied to :—

(1) Stabilisation of Enzymes

The freeze-drying of a crystalline enzyme, catalase, was studied in some detail by Dounce and Howland in 1943 [12]. Twice-crystallised material, suspended in water, was pre-frozen, and then dried. The enzyme activity of the re-suspended dry product was only about 30% of that of the starting material. It was non-crystalline and had a different solubility. The authors considered that the change produced was probably a very mild denaturation. It is possible that this is a result of the irreversible removal of water from within the crystal and further information about the behaviour of crystalline biological materials to freeze-drying must be sought.

Partially-purified bovine testis hyaluronidase was freeze-dried by McCullagh *et al* [38]. The 'unprotected' enzyme gave an unsatisfactory product. Added gelatin had no stabilising action and increased the difficulty of re-constitution, and a partial hydrolysate of gelatin was then used. This polypeptide was easily soluble after drying, and in the presence of this stabilizer the hyaluronidase showed no loss of activity at 10°C for more than six months.

The enzymic activity of crude tissue extracts is, in general, more readily preserved. Doubtless the normal tissue components exert a mutual stabilising action. The author has found that crude rat testis hyaluronidase, and human prostatic acid phosphatase may both be preserved for months by freeze-drying, and storage of the dry product at −2°C.

(2) Preservation of Bacterial and Tissue Culture Media, and of Body Fluids

The use of freeze-drying for the preservation of blood fractions, blood plasma and blood serum has already been described [19 57 58] but the method may be used equally well for the preparation of stable media for tissue culture and bacterial culture [28 29 30 54]. Westfall records the drying of partial protein hydrolysates for nutritional studies [64]. The initial hydrolysate was first concentrated *in vacuo*, and dextrin or starch added to the concentrated solution before pre-freezing. The addition of starch or dextrin, but not of sucrose or ceralose, gave a dry product which was less hygroscopic. Teague *et al*, found that the total nitrogen content of freeze-dried specimens of faeces, urine and milk was the same as that of fresh specimens [62]. Urine was also freeze-dried by Friedgood to give a dry product from which the steroids could be extracted with organic solvents [21]– a much simpler, and more efficient, process than the more usual liquid : liquid urine extractors.

(3) Preservation of Bacteria

Swift used freeze-drying for the preservation of stock cultures of bacteria, especially streptococci and pneumococci [56 59 60]. He recorded the necessity of maintaining the cultures in a frozen state until drying was complete. Bacteria, like viruses and enzymes, vary considerably in their resistance to drying. Stamp was able to preserve *Salmonella paratyphi* B by drying from the liquid state over phosphoric oxide *in vacuo* [55]. Various stabilising compounds were tested. 10% nutrient gelatin (a mixture of gelatin, Lemco broth and peptone) plus 0.1% ascorbic acid gave better protection than horse blood, serum, mucin or polyvinyl alcohol. Many bacteria, *e.g.*, *Chromobact. prodigiosum*, could not be dried in this way but would resist rapid freezing followed by drying. Stamp considered that the gelatin was the protective component in the nutrient gelatin mixture. Naylor and Smith found that cultures of *Serratia marcescens* dried from the liquid state by Stamp's method, and subsequently stored in air over phosphoric oxide, could be protected to a large extent by the addition of ascorbic acid and marmite [40]. By the ' lyophile ' technique, maximum survival of the organism was obtained *in vacuo* in the presence of dextrin, ascorbic acid, thiourea or ammonium chloride in concentration of 2, 0.5, 0.5 or 0.5% respectively at pH 6.0—7.0. Protein-containing protective agents were recommended by Elser and others who successfully freeze-dried bacteria and yeasts [15]. Flosdorf and Kimball used skim milk as a diluent for *H. pertussis* cultures [18].

There is some doubt as to the percentage survival of the bacteria in these dried cultures. Some, such as *Treponema pallidum*, do not survive at all ([42] [63]) and a figure of not more than 5% has been suggested for others [20].

There seem as yet to be no general rules for the successful preservation of all bacterial cultures, and if the culture alone cannot be dried there is an almost unlimited choice of protein-containing compounds and mixtures which may be tested for preservative action. Moreover, the majority of results in this field, and in the field of virus preservation, have been recorded as the alternatives of survival or non-survival, and until recently little quantitative data have been available.

In 1949, Proom and Hemmons published working details for the preservation as desiccates of a collection of more than 1500 strains of bacteria [43]. Various methods of freezing, the efficacy of drying and the effect of storage were tested by viability counts. They concluded that, while it would have been impracticable to keep the strains by sub-culturing, animal passage, selection of colonies, etc., freeze-drying was both practical and efficient. The percentage of organisms which survived was found to be dependent only on the strain—providing that a protective colloid such as gelatin was used, and suitable precautions were taken. Only two factors were held to influence the survival rate after freezing : the species, and the nature of the suspending medium. The protective colloid cannot pass through the cell wall, and it seemed probable therefore that the function of this colloid was to protect the organism against damage by extra-cellular ice-crystals.

Two factors were found to be of critical importance in avoiding high storage loss. The cultures should be as dry as possible, and should be sealed in dry nitrogen or *in vacuo*.

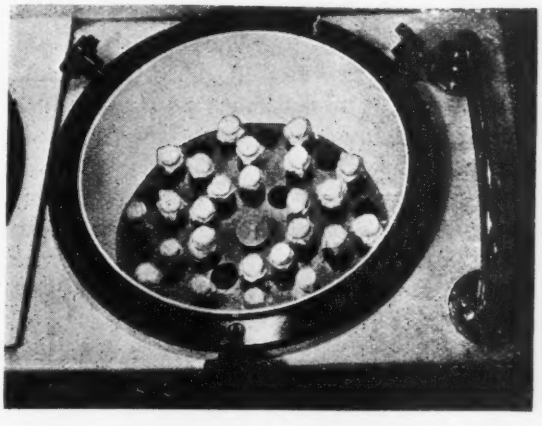

Fig. 2.—Drying Chamber of Freeze-Dryer showing the centrifuge partly loaded with bottles. Each bottle contains tumour-virus suspension and is fitted with a fabric sterility cap.

(4) Preservation of Plant and Animal Viruses

In general, viruses are very difficult to dry, and the dried products, although stable, have often a much lower titre than the fresh materials. Two plant viruses (Y-virus, and Canada streak, of potatoes), have been preserved in 'lyophile' form by Dykstra and du Buy [14]. The viruses were dried in suspension in juice expressed from the plant, and were preserved *in vacuo*, since air inactivated the dry products. Kaiser found that glycerol, and some sugars, protects plant cells against 'cold and drought', and concluded that their action in virus preservation was to decrease the water-loss rather than to lower the freezing-point of the suspension [33].

Many animal viruses have been successfully preserved by freeze-drying infected tissues, or tissue-cultures, with or without addition of protecting substances. Thus, influenza virus was dried in a medium composed of minced chick embryo, Tyrode's solution and 3% gum acacia [50]. Seidentopf and Green stressed the necessity for keeping dried distemper virus either sealed *in vacuo*, or under an inert gas, to prevent oxidation of the lipids [52]. Partially-purified distemper virus may be stabilised for freeze-drying with a protein hydrolysate.

Yellow fever virus was first satisfactorily preserved by drying frozen infected blood or liver in a vacuum desiccator [46]. Bauer and Pickels devised an apparatus for drying large quantities of the concentrated yellow fever virus [1]. They found that the isolated virus, as distinct from the virus *in situ* in infected tissue, was very labile and rapidly became inactive if the protein content of the suspending medium was too low. The virus could not be dried by the 'lyophile' process [16] and Bauer and Pickels suggested that this was a result of the fact, that, as the final stage of desiccation was approached, the cooling effect diminished because of the lowered vapour pressure of the more concentrated material, and of the retarded diffusion of water molecules through an increasingly thick layer of partially-dried preparation. They suggested, without physical evidence, that during the final stage the virus material becomes sufficiently liquid or semi-liquid to allow the concentrated electrolytes to exert their deleterious action upon the virus. In their apparatus the virus material was maintained throughout the drying cycle at a temperature lower than the original freezing-point.

The method adopted by Sawyer (*loc. cit.*) for yellow fever virus was successfully applied to the preservation of the virus of swine influenza and mumps [53 32]. Kitchen preserved the activity of Rift Valley fever virus for 'long periods of time' by freeze-drying it in the serum of origin [34].

Freeze-dried, virus-infected tissues and body fluids were also found to retain virus activity in the case of lymphocytic choriomeningitis and St. Louis encephalitis [66] 'common cold'—where gum acacia exerted a protective action on the virus [11]—experimental measles, lymphogranuloma, sandfly fever, herpes simplex and hog cholera [65 13 45 31 39]. Dysentery bacteriophage could be preserved by concentration of lysates *in vacuo*, followed by freeze-drying [48].

The preservation of purified virus suspensions has always proved to be more difficult, as has already been noted. Rivers and Ward sought to add a material to vaccinia virus suspensions which would (*a*) act as a protective agent, (*b*) add bulk and (*c*) go into solution with ease, and take the virus with it [44]. Egg albumin fulfilled these conditions, but had the grave disadvantage that it was antigenic, and thus unsuitable for injection. 2.5% gum acacia was substituted for the albumin and the dried product retained its activity for over one month at a storage temperature of 37°C. Behrens and Ferguson also found that highly purified vaccinia virus preparations could not be freeze-dried by the normal methods [2]. They studied, as preservatives, 0.1% and 1.0% peptone, 25% and 50% glycerol, 10% inactivated rabbit serum, 0.1% gelatin, isotonic glucose, and 1 : 1000 and 1 : 2000 cysteine hydrochloride. Of these, 1% peptone and 0.1% gelatin gave the best protection against inactivation.

(5) Preservation of Tumour-Producing Viruses

It has long been known that tumour tissue, both sarcomatous and carcinomatous, can be kept frozen

Vol. 1 1951

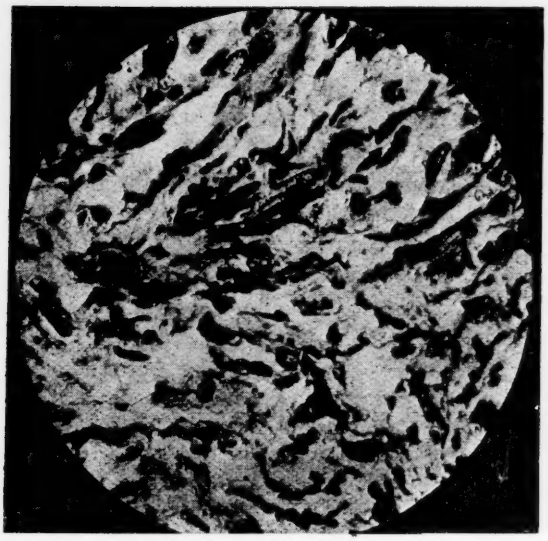

Fig. 3.—Rous Sarcoma. Stained section. Magnification: x 120.

for considerable periods at low temperatures, *i.e.*, -10°C to -79°C, and still retain the power to start fresh tumours after thawing, and inoculation into the appropriate animal. The possibility has always remained that the tumour cells themselves do not survive the freezing, and that the new tumour is produced by an agent within the cells, and able to survive treatment which the cells cannot. Any tumour which could be transmitted in such a 'cell-free' manner would fall within the group of known virus-induced tumours: the chicken sarcomata, the Shope rabbit papilloma, mouse mammary carcinoma and Lucké's leopard frog adenocarcinoma. Freezing-and-thawing, alone, is an unreliable method of ensuring tumour cell death. Some tumours, and many normal tissues, will survive a number of cycles of alternate freezing and thawing without the death of all the cells [3 4 35]. Mouse sarcomas, such as C 48 (methylcholanthrene-induced in inbred C 57 mice), R3βR.T. and C3HR.T., (sarcomatous transformations from carcinomata) have been found by Gye and his associates to resist all attempts at cell-free transmission and, moreover, they not only resisted freezing and thawing, but also such procedures as fine mincing and dispersion, dilution with glucose, or with cysteine, buffering to pH 6, storage in glycerol, and treatment with distilled water, which had been shown to kill normal mouse embryo cells [37]. Gye, Begg, Mann and Craigie were aware that all these methods of approach are open to the objection that a few cells might survive. They, therefore, proceeded to 'dry the refrigerated tissue completely to dust *in vacuo* at a temperature of -25°C' [25].

The apparatus used was designed by Craigie [9]. The minced tumour tissue, suspended in 5.3% dextrose (which favours the survival of tumour activity in freezing-and-thawing experiments) was held at a temperature of -22° to -30°C, and the sublimed water vapour condensed at -79°C. A specially designed vapour flow indicator was the only means available of following the course of drying. It consisted of a light vane suspended freely from a pivot so that it was displaced by the current of vapour leaving the drying specimen. The vertical component of the weight of this vane, at a displacement of 1° from the rest position, was 6.5×10^{-2} mg. After the vane had reached zero during the drying cycle, pumping, it is stated, was continued for a further half or one hour to be absolutely certain of the removal of the last traces of water.

The actual material dried in this apparatus was C 48 sarcoma and R3βR.T. sarcoma, pre-frozen for 28-100 days at -79°C. The dry products were immediately reconstituted and injected into the test animals. Tumours were produced.

The conclusive proof that a virus aetiology of these and, by inference, of all tumours has been established must await conclusive evidence that the desiccates were really dry. No attempt was made by Gye and his group (*loc. cit.*) to show that the dried tumour could be stored or that it was incapable of producing growth in tissue culture, where such growth would demonstrate the presence of living cells.

The Rous chicken sarcoma No. 1 virus, which the author has been studying with his colleague Dr.

J. G. Carr, can certainly be preserved by desiccation of tumour tissue [31 36 59]. Knox recorded that he had successfully dried Berkefeld N filtrates of fresh tumour material (Rous sarcoma, Fujinami myxosarcoma) though with some attendant loss of activity.

The Rous sarcoma virus has been obtained in a purified state from fresh tumour tissue by a combination of differential centrifugation and enzymic digestion (Carr and Harris, in press). A reliable assay method has been devised, and it has thus been possible to study quantitatively the effects of different procedures on the stability of the virus to freeze-drying and to storage in the dried state.

A commercial centrifugal freeze-drier has been most successfully used throughout the work, and primary drying alone has been found to be adequate. The procedure has generally been to re-suspend the virus pellets (from the centrifuge) in 10 c.c. aliquots of the medium under investigation, and to titrate the the fresh virus before freeze-drying in McCartney bottles. A drying time of thirty-six hours has been adopted, and the dried virus has been stored in the sealed, air-filled bottles at −2°C. The dry virus material has been re-suspended to the initial volume, and cleared of aggregated material by centrifugation, before titration.

The results obtained confirm the data for other viruses in that, as purification of the virus proceeds, it becomes increasingly unstable to freeze-drying. The Table summarizes the essential data. From a

THE EFFECT OF THE SUSPENDING MEDIUM ON THE PRESERVATION BY FREEZE-DRYING OF THE VIRUS OF CHICKEN TUMOUR 1

Preliminary Purification Stages	Suspending Medium for Freeze-Drying	Titre	
		Fresh	Dry
2	M/100 Disodium hydrogen phosphate	$> 10^{-5}$	nil
2	Gelatin (1 mg./c.c.)	do.	10^{-2}
2	Egg albumin (1 mg./c.c.)	do.	10^{-2}
2	Soluble starch (B. D. H. Analar) (125 mg.%)	10^{-7}	10^{-2}
2	Cysteine-HCl (adj. to pH 7.2) (125 mg.%)	do.	10^{-2}
2	Dextrose (125 mg.%)	do.	2×10^{-5}
2	Lemco nutrient broth (Lemco), pH 7.4	do.	2×10^{-7}
2	Water	do.	10^{-2}
3	Water	2×10^{-8}	10^{-1}
3	Dextrose, 5% (adj. to pH 7.2)	do.	10^{-1}
3	Dextrose, 3% (do.)	do.	10^{-2}
3	Dextrose, 1% (do.)	do.	$> 10^{-2}$
2	Chapoteaut Peptone 1.25%	10^{-8}	nil
2	Lemco : 5% Dextrose, 1 : 1	do.	10^{-3}
2	*Gelatin hydrolysate, pH 7.2	10^{-5}	10^{-2}
2	Lemco, pH 7.4	10^{-5}	10^{-5}
2	Lemco, pH 8.5	10^{-5}	10^{-6}
2	B.D.H. Peptone, 1.25%	do.	10^{-4}
2	B.A.L. glucoside, 1 : 1000, pH 7.2	do.	10^{-2}
2	1 : 2 Guinea-pig serum : water	do.	10^{-4}
2	**Protein hydrolysate	do.	10^{-4}
2	***Cellofas A, 0.3%	do.	10^{-1}

* Knox Gelatine Protein Products Inc., N.Y., U.S.A.
** By courtesy of Dr. F. V. Linggood. Burroughs Wellcome.
*** By courtesy of I.C.I., Ltd.

virus *concentrate* having an initial activity of 10^7 per c.c., (1 c.c. representing the yield of virus from 1 g. fresh tumour), it has been possible to recover 50-100% of the original activity after re-hydration, even after storage for five months. This is highly satisfactory from a virus preparative point of view, but unsatisfactory in that the mode of action of the protective substances used is still unknown. Schade has suggested, on the basis of his work with bacteriophage, that lipid anti-oxidants such as freshly prepared lecithin, are good protective agents ([49]). He found that bacterial lysates lost 99.9999% of their original phage count on freeze-drying (count reduced from 1×10^9 particles per c.c. to 1×10^3). Addition, before freezing, of Difco meat extract took the final count up to 1×10^5. Polysaccharides, in general, reversed this trend and glycogen reduced the count to zero. Schade found that extracts of fresh brain, kidney or pancreas gave complete protection and, after trying egg yolk and skim milk, he concluded that lipids such as ovolecithin were the essential agents [48] [49].

Vol. 1 1951

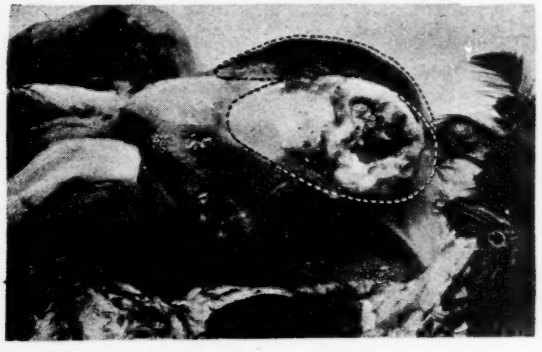

Fig. 4.—Rous Sarcoma growing in both breasts of a 9-week old chicken.

Serum has frequently been used as a diluent for virus suspension, but is unsatisfactory because of the presence of non-specific virus-inactivating sub-

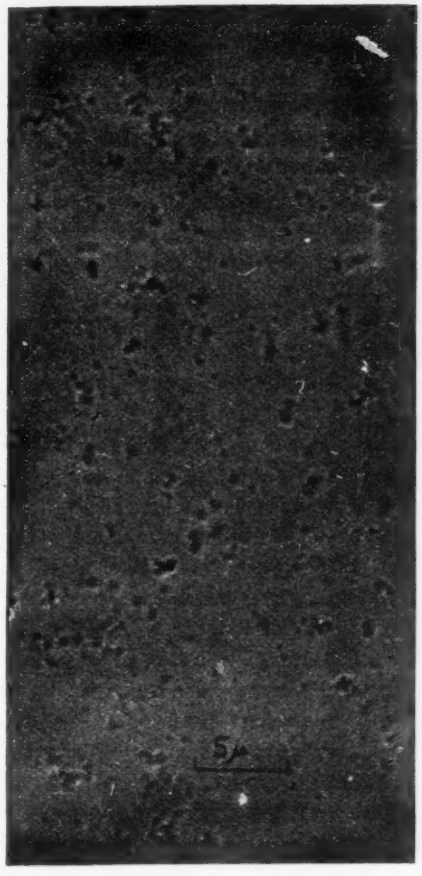

Fig. 5.—Separated particles from Rous Fowl Sarcoma. Photomicrograph taken with U.V. radiation λ-257 mμ. See R. J. King and E. M. F. Roe—Reference 33a.

stances. Broth has been used, or skim milk, or, more recently, buffered bovine plasma albumin [10]. It may be, therefore, that one function of a protective agent is to reduce the tendency of viruses to aggregate. It is reasonable to conclude that the optimum protecting agent should have the properties :—(*a*) be ultimately readily separable from the virus, (*b*) provide bulk to the product, (*c*) prevent surface denaturation, (*d*) prevent virus aggregation, (*e*) be non-hygroscopic, (*f*) act as an anti-oxidant and (*g*) provide an optimum reduction of the freezing temperature of the mixture [33] [44] [49] [64].

TECHNICAL PROBLEMS STILL TO BE SOLVED

The drying procedure for the Rous virus has been standardised as far as possible, but much greater control is obviously necessary if the optimum conditions for the preservation of viruses are ever to be realised.

The **rate of freezing** of the virus suspension in different media must be determinable. The rate of freezing has been found to be of vital importance in the resistance of cells to freezing-and-thawing and, if viruses contain internally-bound water, may well be equally critical for them [3].

The **temperature of drying** is a second important factor. Samples of broth, for example, may dry well at -30°C, though in some cases it has been necessary to go as low as -60°C to obtain perfect desiccation [23]. This may be the reason why some viruses do not survive drying in some media [1].

The **final moisture content** has been found to be a critical factor in the storage of freeze-dried blood plasma and serum. The American practice has been to use single-stage drying to a final moisture content of 0.2 to 0.5% and in no case greater than 1% [20]. British practice produces a first-stage product with a water content of about 0.4%, which is then secondarily dried over phosphoric oxide [23]. There seem to be no comparable figures in the literature for the preservation of viruses. Moreover, a rapid and readily reproducible method of determining the water content of very small amounts of such materials as dried virus is urgently required. The vacuum oven method, and the Karl-Fischer technique both destroy the material under investigation and the alternative of drying over a chemical desiccant and weighing *ad infinitum* is both tedious and inaccurate for very small quantities [17].

Lastly, it may be observed that cells and tissue sections can apparently be dried without salt migration, only if they are quickly frozen to produce a glass ice, and then if the subsequent dehydration is accomplished at temperatures below about −55°C. Ordinary freeze-drying temperatures no doubt exceed this and cannot be expected, therefore, to immobilise inorganic ions [1]. Pease prepared aqueous films of purified myosin dissolved in potassium chloride solutions of 0.3 to 1.0 M [41]. The films were frozen in liquid air and dehydrated at a constant temperature of −72°C. In the electron microscope, no trace of salt deposition could be found. Pease suggested that the ions might have evaporated from the regressing glass ice surface during dehydration. It will be interesting to know whether this observation can be confirmed, and, if so, whether similar ionic migrations and evaporations may be expected to occur within the structure of bacteria and virus particles.

The author wishes to express his indebtedness to his colleague, Dr. J. G. Carr, F.R.S.E., and to thank Dr. E. M. F. Roe and Mr. R. J. King for permission to use Figure 5, and Mr. F. E. Speed for the preparation of Figures 2 and 4.

Vol. 1 1951

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Vol 1 195

The production, properties and uses of THIN FILMS CONDENSED IN VACUO

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Summary

THE STUDY of the properties of thin films deposited *in vacuo* has grown considerably in recent years. The vacuum equipment involves special design considerations, three major aspects of which are discussed–(1) Evaporation pressure (2) Pumping speed and ultimate oil vapour pressures, and (3) the use of discharge techniques for the cleaning of contaminated supports. Applications are fully reviewed together with results of research work on structure and physical characteristics of films

Sommaire

L'ETUDE DES PROPRIETES et de la structure des couches minces intéresse les hommes de science de plus en plus. Le développement des appareils pour le revêtement sous vide nécessite des considérations spéciales qui sont discutées par l'auteur sous trois différents aspects : la pression requise, le débit des pompes et le nettoyage de la surface de base par un bombardément ionique. Les progrés récents ainsi que les derniers résultats obtenus dans l'étude des proprietés des dépôts sont passés en revue.

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INTRODUCTION

THE PREPARATION of solid thin films by the technique of vacuum evaporation and, in certain applications, by cathodic sputtering is becoming of increasing importance for research work. Evaporated films are now employed in the preparation of specimens for eleetron microscopy and electron diffraction studies, for multiple beam interferometry, anti-reflection coatings on glass and other techniques.

The laboratory study and application of evaporated films has been stimulated and widened because of the comparative ease with which it is now possible to obtain low pressures—of the order of 10^{-5}mm. Hg—in kinetic pumping systems. In addition, the processing methods required for depositing metals and their compounds on to a variety of substrates have been worked out more fully. The growing application of vacuum coating techniques is signified by the amount of scientific and technical literature published on this subject and events as, for instance, the recent conferences on thin films held in France and America [1] [2].

* MS. received in August, 1950

Many laboratories have, in the past, constructed their own vacuum evaporation equipment. In certain cases, this was necessary because of the low order of pressure required. Measurements of optical constants, for instance, demand the lowest pressure obtainable to avoid film contamination by oxidation. In other cases, laboratory construction was dictated by economy considerations or the lack of industrially produced equipment catering for laboratory needs. It is the purpose of this article to deal with the factors governing the design and development of laboratory coating units, to discuss some of their applications and to survey recent research work on the properties and structures of thin evaporated films.

'Vacuum Evaporation' consists in raising the temperature of a thermally stable material *in vacuo* so that volatilization takes place and the emitted vapour is condensed on to surfaces surrounding the evaporating source. The gas pressure in the chamber must be decreased to a point where oxidation is kept to a minimum, *i.e.*, in the normal case to 10^{-4} mm. Hg or lower. In this pressure region, the mean free path of the residual gas molecules is some 50 cm.

Thus, providing the distance between vapour source and receiving surface is much less than 50 cm., the majority of the vapour molecules traverse the intervening space without colliding with gas molecules. Due to the limited number of collisions the vapour molecules tend to travel along straight paths and, therefore, are often referred to as ' Molecular Rays '.

The number I (in %) of emitted molecules which arrive at the condensing surface without experiencing collisions can be calculated from the following expression :—

$$I = I_o \exp\left(-\frac{d}{L_p}\right) \qquad (1)$$

where I_o = the intensity of the vapour at the emitting source.
d = the distance between emitting source and receiving surface.
L_p = the mean free path of the vapour molecules in the residual gas at a given pressure in the chamber.

The formula shows that a higher pressure can be tolerated if d is reduced to a minimum. In practice, however, the emitting surface must be kept at a certain distance from the condensing surface so that heat radiation from the source does not damage the film. Further, most emitting sources have a spherical-like distribution and this, in its turn, controls the position of the source in relation to the condensing surface in accordance with the film distribution desired [3].

Under conditions of high vacuum and where the vapour pressure of the substance being evaporated is low, *i.e.*, less than 1 mm.Hg, the vapour molecules can be assumed to leave the emitting surface relatively unimpeded. The mass of substance removed as vapour in unit time, from the emitting surface, is then the same as the rate of evaporation of a substance in equilibrium with its saturated vapour, as shown by Langmuir [4]. For a substance to be in equilibrium with its saturated vapour the rate at which molecules evaporate from the surface must be equal to the rate at which they are condensed. The formula given below is derived from the rate at which molecules strike a surface at a given pressure, if only a fraction *a* of these molecules on impact are condensed then for equilibrium the evaporation rate will be reduced by a similar amount. The ' accommodation coefficient ' *a* is usually assumed to be equal to unity in the case of metals.

The mass evaporated is given by :—

$$5.85 \times 10^{-2}\ a\ P\sqrt{\frac{M}{T}}\ \text{g.cm.}^{-2}\ \text{sec.}^{-1} \qquad (2)$$

where P = the vapour pressure of the material in mm. Hg at the temperature of T°abs.
M = gram. mol of the substance being evaporated.
a = accommodation coefficient.

Useful vapour pressure-temperature data for metals, many of which are used in the evaporation technique, has been prepared by Kelly [5], Law [6] and Dushman [7]. Most metals, some of their oxides and various salts, *e.g.*, fluorides, are sufficiently volatile to be used for preparing thin films. The deposition of alloys is more complicated, in particular, where the constituents possess widely different vapour pressures, *e.g.*, brass, but a method has been reported whereby the alloy in granular form is brought into sudden contact with a heated surface so that distillation cannot take place [8].

The distribution of condensed films has been dealt with in a simple treatment by Strong [3] and various types of heaters from which the material can be vaporized, have been investigated [9] [10].

Usually, the heaters are made of high-melting point low-vapour pressure metals such as tungsten, molybdenum or tantalum. Ordinarily resistance-heating is employed to raise the source temperature. However, electronic bombardment and high frequency-heating have also been used. Ceramic crucibles, *e.g.*, alumina heated externally, can only be used with limitations because of their poor thermal conductivity, low resistance to heat shock and possible reduction of the oxide by the metal being vaporised. The useful life of a heater is normally determined by the degree of alloying which takes place with the charge and its fragility

Vol. 1 1951

due to embrittlement after degassing. Platinum for example requires a very high temperature for volatilization and reduces the life of the heater appreciably due to alloying.

To facilitate the evaporation of large quantities of metal further development of crucible design is still required. The author has experimented on a laboratory scale with evaporating large quantities of metals from resistance-heated carbon crucibles. In the case of aluminium a dense grade of carbon must be used to prevent aluminium from diffusing through the crucible wall. A further difficulty arises from the high ' wetting ' power of the molten aluminium as a result of which the solidified aluminium adheres so strongly to the crucible that contraction on cooling can break the crucible. The aluminium and carbon react, forming a carbide, which gradually leads to disintegration of the crucible.

Although the cathodic sputtering process is of more limited use than vacuum evaporation for the production of thin films, it is used for the deposition of precious metals and has recently been employed by electron microscopists for the etching of metallurgical specimens [11]. The determination of the deposition rate in terms of the applied discharge voltage and current density, etc. is largely empirical. The author has found the following formula of value in the design of sputtering plant employing large plane cathodes with negligible edge losses :—

$$S = \frac{(Va - Vc)\, iK}{d\, p} \qquad (3)$$

where S = the deposition rate (mass deposited /sec.).
Va = applied voltage.
Vc = a constant (approx. 500V).
i = the current density mA/cms^2.
d = the distance separating cathode from receiving surfaces.
p = the pressure of the discharge gas at constant temperature.
K = a constant dependent on cathode material, discharge gas and cathode arrangement.

THE DESIGN AND OPERATION OF VACUUM EVAPORATION PLANT

In designing evaporation plant, stress must be placed on the provision of high speed pumping equipment compatible with economy whilst, at the same time, protecting the workchamber against direct entry of vapours from the diffusion pump fluid. In kinetic systems the highest pumping speeds are usually required in the low pressure region of approximately one micron or less, where gases and vapours are more freely liberated from surfaces, and for the rapid removal of gases released from the substance being vaporized. Also a certain amount of leakage from vacuum seals must be envisaged, This, together with the degassing just mentioned, gives rise to an equilibrium pressure depending on the speed of exhaustion.

Degassing in Vacuum Systems

Sometimes it is possible to accelerate degassing in parts of a continuously pumped system by raising the temperature of the workchamber and its associated fixtures, but such measures cannot be applied to all parts of the apparatus, for instance, the diffusion pump tubulations, especially not when a metal system is employed and the vacuum seals are heat-sensitive.

The use of Silastic rubber shows promise in overcoming the deficiency of vacuum seals in that respect but still it is obvious that very high speeds are necessary, if low pressures are to be obtained in a reasonable pumping time.

It is well known that degassed surfaces on exposure to the atmosphere can take an appreciable time to re-adsorb gases and it is, therefore, important, to keep such exposure to a minimum. Weingartner [12] who has measured the pump-down time of a 4 ft. diameter stainless steel chamber found that below one micron the exhaustion time was dependent upon length of atmospheric exposure and was thus the dominating pressure range of degassing. Many coating laboratories fitted with air conditioning and humidity control claim that exhaustion time remains

constant and the condensed films are improved.

It is considered by a number of workers that a contaminated system can be exhausted more rapidly by allowing bursts of gas into the vacuum chamber, a practice that the author has never found effective. Recently Chupirin[13] published details of the 'rinsing' method and claimed that the process speeded up degassing. He submits that the degassing equilibrium is altered by the migration of oxygen into the surface contaminants. To minimise re-adsorption it is useful to fit an isolation valve between diffusion pump and chamber so that when the chamber vacuum is broken the pumping system remains under vacuum.

Rate of Exhaustion

It is usually impractical to calculate for a given apparatus the pumping speed required in the low pressure region, largely because insufficient data exists on the degassing rates of the various constructional materials. Where such data exists its use other than qualitative is further complicated by a lack of knowledge of the adsorbed gases, surface area, etc. Modern methods of design and leak detection enable definite standards of vacuum tightness to be achieved: thus the pumping speed required to handle leakage can be calculated, but the pumping speed needed to cope with degassing must usually be arrived at from practical experience.

The rotary pump employed should have sufficient speed to handle the maximum through-put of the diffusion pump. Since the volumetric efficiency of a rotary pump decreases at lower pressures, it is obviously economical to employ diffusion pumps with high backing pressure performances. This will reduce the size of the forepump required and permit the diffusion pump to be brought into operation at an early stage of the pumping cycle. Until recently, oil diffusion pumps with a maximum backing pressure of only 0.15 mm.Hg were available but it is now possible to obtain pumps with a backing pressure of 0.35 mm.Hg - 0.5 mm.Hg.

A high backing pressure also allows the pumping system to continue to function with a significant deterioration in rotary pump performance as for example when the rotary pump oil becomes contaminated with vapours removed from volatile materials in the coating chamber.

Contaminating Vapours

Care must be taken to see that the diffusion pump is adequately baffled against direct entry of vapour molecules into the vacuum chamber. For oil diffusion pumps a partial pressure of oil vapour in the chamber must be accepted but a water-cooled baffle, inserted between pump and chamber, will keep it down to a satisfactory quantity. The cooled walls of the baffle condense the oil vapour so that the pressure of the oil vapour in the vacuum chamber is not greater than its saturation pressure at the cooling temperature provided.

If it is desired to raise the temperature of the substrate in the vacuum chamber, heat radiation should not be allowed to penetrate into the diffusion pump baffle chamber. Also a drying agent, such as phosphorus pentoxide, should be employed. This is especially mentioned as it is not always realised that the evaporation rates of certain metals can be accelerated in the presence of water vapour. Tungsten, for instance, which is normally used as the heater material in the evaporation source can be evaporated at rates far exceeding those calculated from its vapour pressure characteristics and thus lead to contamination of the deposited film [14].

Measurement of Ultimate Pressure

A complication in all vacuum work is the measurement of pressures in the low pressure region and conflicting results have been obtained in many cases because of erroneous methods of determining pressure. Where industrial applications are concerned, for instance, the production of front-surface mirrors or anti-reflection coatings, the Penning cold cathode type of ionization gauge is sufficiently sensitive to both gases and vapours and quite satisfactory: but it cannot be used for accurate pressure measurements due to the different ionization coefficient of the unknown mixture of gases in the chamber [15]. Thus, if a study is to be made of the condensed film itself, the properties of which can depend to a large extent on the nature and pressure

Vol. 1 1951

of the residual gases, the ionization gauge must be used with caution. It is not possible to discuss here the relative merits of the various gauges except to state that the Knudsen gauge would appear to be the most suitable for this kind of research work. It is true that, for the Knudsen gauge as well, the composition of the residual gases remains unknown but the gauge does accurately measure the total pressure of gases and vapours.

Low Pressure Discharge Cleaning

It is usual to pass a gaseous electrical discharge in the evaporation chamber before deposition is commenced. It has been found that the bombardment of a surface by ions formed in the discharge removes surface contaminants which remain after chemical cleaning and assists in a general clean-up of the vacuum chamber.

The onset of the discharge is usually accompanied by a rise in pressure, brought about by ionic bombardment of the chamber walls, etc. Whereas in sealed off tubes the gas evolved is gradually re-adsorbed by the walls and electrodes, in a continuously pumped chamber this gas is largely removed by the pumps. If the discharge is to continue, a supply of gas must be admitted to the vacuum chamber *via* a needle valve. Usually the initial colour of the discharge is blue, denoting the presence of water vapour which is quickly removed by the 'rinsing' action of the air admitted. The colour then changes to that for dry air. A discharge can, therefore, be invaluable for assisting in the clean up of a vacuum system.

It has been common practice to earth one side of the high tension supply so that the baseplate supporting the diffusion pump operates as one discharge electrode whilst the second electrode is led into the chamber *via* an insulated seal. This is not a satisfactory arrangement. The high tension supply should not be earthed when alternating current is used, because the pumping aperture of a diffusion pump is usually large enough to allow a discharge to be maintained in its throat for the period the earthed electrode is negative. This can result in the performance of the oil baffle being affected and oil decomposition products being formed in the chamber.

Vacuum seals with greased surfaces must not be placed in regions which the glow discharge can reach but the use of modern 'trapped rubber' seals renders this precaution unnecessary. The chamber fittings, where possible, should be constructed of aluminium and the high tension electrodes must be made from this metal to prevent sputtering. The electrodes should also be shielded against the deposition of evaporated metal which would sputter during subsequent glow discharge periods.

Shown in Fig. 1 is an electrode arrangement used for bombardment of lenses, etc., prior to anti-reflection coating. Metal substrate surfaces can be effectively discharge-cleaned if they are connected to one side of the high tension supply. However, in this case, it is preferable to rectify the supply and to

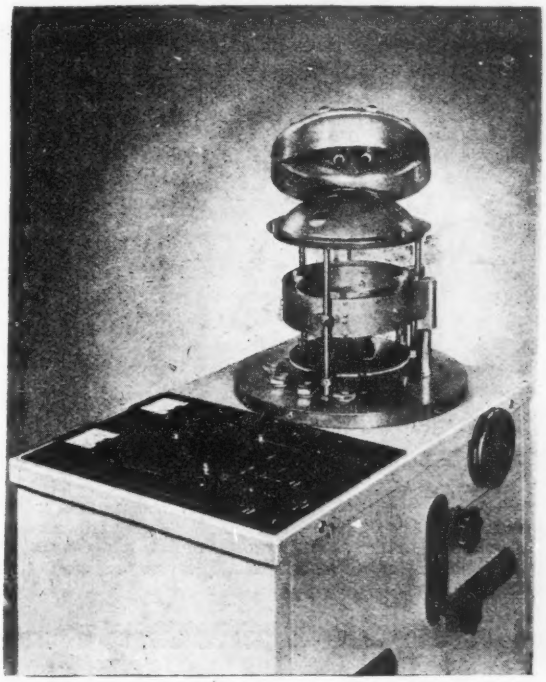

Fig. 1. Vacuum Evaporation Plant used for the deposition of interference films, etc. The discharge electrodes are two annular rings fitted to a tripod which also supports a spherical workholder. The radiant heater, arranged above the workholder, can be used for raising the substrate temperature.

connect the substrate as the cathode. To prevent oxidation, hydrogen or an inert gas, such as argon, should be leaked into the chamber after the system has been allowed to degas at a low pressure. The nature of the discharge gas is undoubtedly of importance when cleaning glass substrates. The author observed that the adhesion of aluminium films on glass substrates was often reduced if the bombardment was carried out in argon or hydrogen instead of air.

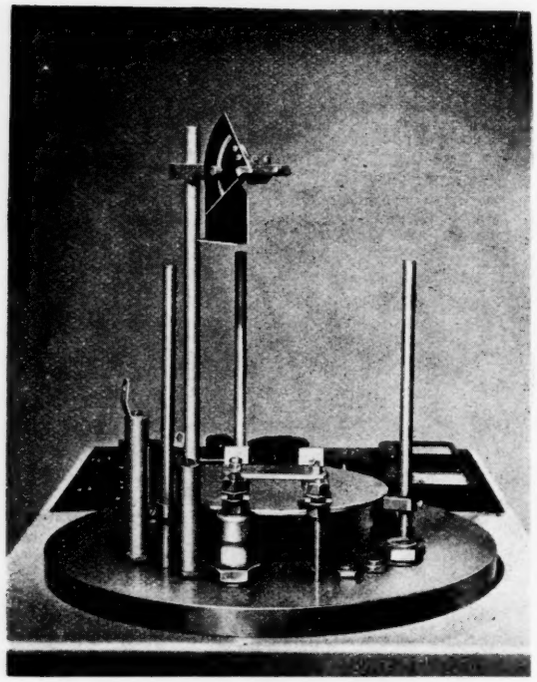

Fig. 2. Shadow casting jig for the preparation of electron microscope specimens. The angle of inclination of specimen surfaces to the vapour source can be adjusted and read on the protractor scale, 4^0 is the preferred angle of inclination.

APPLICATIONS OF THIN FILMS IN THE LABORATORY

Electron Microscopy

A technique which improves the image detail of specimens examined in the electron microscope by applying a thin metallic film to the specimen surface has now gained great importance. The metallic film is deposited by evaporation and the specimen arranged in such a manner that it receives the molecular rays at an oblique angle. Thus a metallic ' shadow ' is cast of any particle which is elevated above the surface. It is possible to calculate the elevation of various parts of the specimen from a measurement of the shadow length and shadowing angle. Fig. 2 shows a Shadow Casting Jig fitted to an evaporation plant and Fig. 3 a shadow-cast specimen.

The metal film improves the detail detectable because the electron scattering power of certain metals is much greater than that of biological material. The different thickness of metal deposited on the specimen surface brought about by the variation of the receiving angle produce variations in the scattering of the electron beam. A photograph of such a specimen gives the impression of being illuminated by oblique lighting (Fig. 3). Recently a method has been described whereby the specimen is rotated whilst undergoing ' shadowing '. This, it is claimed, increases the overall definition of the specimen still further [16]. Specimen supporting films of materials with a low electron-scattering power, *i.e.*, silica, silicon monoxide and beryllium, are also prepared by evaporation.

The granulation of the metallic film on the specimen has been studied together with the adherence of evaporated films to glass. The latter is of importance when the specimen is prepared and shadowed on glass before being backed with collodion and stripped from the glass support. It was observed that materials which oxidise readily have a high adherence to glass [17].*

A review of recent work has been made by Drummond [18] and a series of papers have been published dealing with applications of electron microscopy in metallurgy [19].

In connection with specimen preparation, Grivet mentions the use of the sputtering technique for the cathodic etching of metals in a glow discharge [19].

* The Summer School on electron microscopy held at Cambridge University has prepared a report for the use of students on shadow casting technique.

Vol. 1 1951

Shown in Fig. 4 is a coating plant fitted with a sputtering attachment, the cathode electrode, normally used for deposition, can be replaced by a metal specimen when employed for etching.

Light Microscopy

The benefits of shadow casting can be extended to optical microscopy. For instance, Wyckhoff and Scott describe the preparation of aluminium shadow-cast replicas of tooth surfaces for examination under an ordinary microscope [20]. Wyckhoff and Williams report on the production of shadowed collodion replicas taken from machined surfaces [21].

RESEARCH INTO THE PROPERTIES AND FORMATION OF THIN FILMS

Optical Properties

The alteration of transmissivity of thin metallic films after deposition and on exposure to air is well known and due allowance must be made for this when preparing certain films of a fixed density [22]. The author has observed the transmission changes for thin aluminium films, the colour of which can alter from a bluish grey to neutral indicating formation of oxide. Alterations of a chemical nature may also take place in a substance during deposition, *e.g.*, the author has found when sputtering iron in oxygen that the deposited film, which is extremely hard, closely resembles in appearance ferric oxide, whereas evaporated ferric oxide appears to break down to a lower oxide and is almost neutral in colour.

High refractive index and low absorption layers have been formed on glass by oxidising condensed metal films and by cathodic sputtering in an oxygen atmosphere [23]. A titanium dioxide film, for instance, produced in these experiments measured 1% absorption, 40% reflectivity and a mean refractive index of 2.6 in the visible spectrum.

The reflectivity of metal films deposited on glass substrates has been investigated by many workers. The greater thickness of opaque mirror coatings (500 Å and more) ensures greater stability of the optical properties of the film. Sabine [24] measured the performance of a number of metals in the ultra-violet and visible region and Bueche [25] in the near infra-red.

Bateson [26] recorded, as the result of a long term experiment, the average reflectivity in the visible region of aluminium and rhodium mirrors which had been prepared under controlled manufacturing conditions.

Aluminium is of special interest because of its usefulness as a front-surface reflector due to its non-tarnishable properties, a great advantage compared with silver.

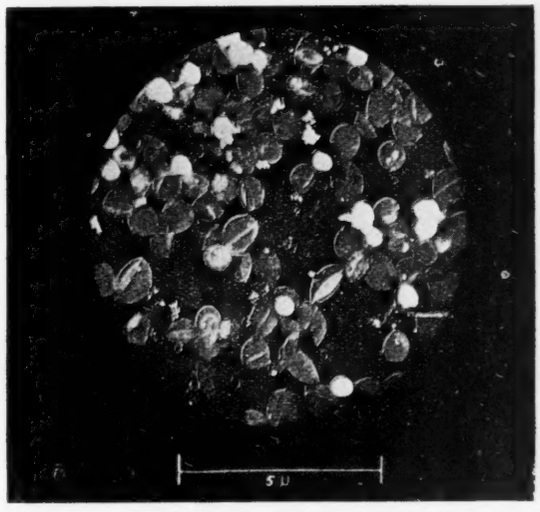

Fig. 3. Palladium shadowed staphylococcus Aureus shells (by courtesy of the Medical Research Council).

Hass [27] measured the growth of the natural oxide on aluminium films after exposure to the atmosphere, the oxide is amorphous and reaches a maximum thickness of approximately 45 Å after one month.

Scott and Hass [28] published the reflectivity characteristics of front-surface aluminium mirrors protected against wear and corrosion with a layer of silicon monoxide. They state that an aluminium film coated with a protective layer about 1500 A thick shows a visual reflectivity of 89%, *i.e.*, only 1% less than that of the unprotected film. Comparison of these reflectivity curves with those for unprotected

aluminium films, shows, however, that the protective film reduces the reflectivity by as much as 15% at a wavelength of 4,000 Å with the maximum reflectively at 5,000-6,000 Å. The author found that aluminium mirrors protected with an SiO layer of the thickness quoted above appear greenish when viewed at normal incidence. The deposition of silicon monoxide is usually detected by observing colour changes on the mirror at glancing incidence.

If SiO is evaporated at temperatures, greater than 1350°C, the deposited film contains silicon and the mirror has a yellowish appearance. In a private communication from Hass [29] it is stated that " Since the electron diffraction pattern of extremely fast evaporated material shows besides the intense halo neither a ring of silicon nor of silica, it must be assumed that a uniform solution of silicon and silica having a ratio silicon to oxygen greater than one, can exist". Unlike silica, SiO is very absorbent in the ultra violet but this disappears as the film is oxidised. Both the ultra violet absorption and refractive index of the film in the visible region is increased by rapid evaporation. Titanium dioxide is of interest for protecting mirrors because of its low absorption and extreme hardness but the author found great difficulty in evaporating this material directly because of decomposition. By oxidising a titanium film deposited on to a previously protected SiO-aluminium mirror Hass found it possible to improve hardness and increase the light reflection from 90% to 96%. With a similar protective combination rhodium reflectively can be increased from 77% to 90%.

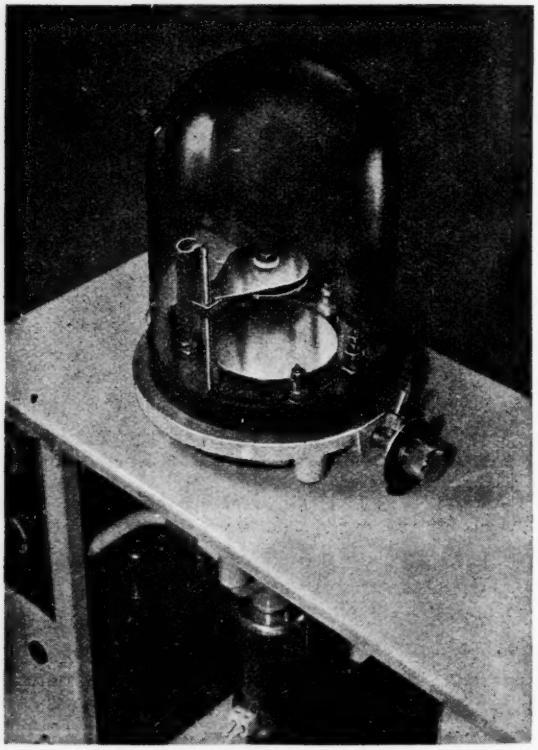

Fig. 4. A small coating plant fitted with sputtering electrodes. The cathode is suspended from the anode by means of an insulator. The small gap between the electrodes suppresses discharges and prevents sputtering from sides of the cathode remote from the receiving surface. When used for etching metallurgical specimens the cathode is removed and the specimen connected in place.

Silica can be vaporised directly but must be evaporated slowly to avoid decomposition as in the case of silicon monoxide, Hass [28] and others claim that evaporation of silica produces flaky deposits but this was not confirmed in experiments conducted by the author. Weinrich [30] describes a method which greatly simplifies the practical problem of heating and melting silica. Hass [31] in a recent article deals with the natural oxide formation on aluminium mirrors and describes a method of increasing the thickness of the protective oxide layer by electrolytic anodic treatment. McRae [32] found that burnishing aluminium mirrors in the presence of water increases considerably the wear resisting properties of aluminium. To what extent the burnishing either changes the aluminium structure or increases the oxide film is not clear but the author has found this simple treatment to be most effective.

It has been common practice to deposit a chromium film on glass prior to aluminising because of an apparent increase in the hardness obtained in the aluminium film. Beeching [33] has studied aluminium films prepared on chromium and glass substrates but could find no difference in their structure. The

chromium layer is not of great importance, however, if a protective layer is deposited or the natural aluminium oxide film increased. Aluminium resistance to salt water corrosion is not very strong but is further weakened if the chromium layer is present [34].

Numerous references exist on the properties of multi-layer interference filters, various types of which are now being made available commercially. The filters can be divided into two main groups (*a*) transmission of narrow spectral bands [35 36] and (*b*) beam splitters [37]. The reflectivity and transmission properties of the latter are complementary and their absorption negligible.

For the purpose of correcting spherical aberration Scherby [38] has used thick laminated films of lithium fluoride and collodion on glass. The laminated collodion film was introduced to prevent the lithium fluoride from forming a granular structure.

Electrical Properties

There is a difference between the resistance properties of very thin films (below 100 Å thick) and bulk metals: the resistivity of the former is usually very much greater than that of the latter and below a critical thickness most metals possess a negative temperature coefficient.

The high resistivity of very thin films is attributed to the film thickness being comparable to the mean free path of the conduction electrons plus the fact that very thin films have a granulated structure and are therefore somewhat discontinuous [39]. Granulation in evaporated silver films can prevent conduction completely even at thicknesses of 400 Å if the substrate temperature is maintained at 300°C. during deposition [40]. The resistivity may continue to alter after deposition and may increase due to aggregation or decrease due to crystallisation, see Itterbeeck [41]. Harris [42] has recently measured the decrease in resistivity of antimony films 50 Å thick. Wilkinson and Birks [43] found that gold films condensed at liquid air temperature decrease in resistance when raised to room temperature. They found that both the initial resistivity and decrease in resistance was greatest for films deposited rapidly. They conclude that the resistance changes are due to defects in the crystal lattice which disappear as the temperature increases. At higher temperatures, up to 300°C. it is not clear whether the decrease of resistance is due to recrystallisation or release of strain.

The film resistivity is also effected by adsorption and oxidation when exposed to gases at higher pressures [44].

In the case of some metals, *e.g.*, chromium, silver, it is claimed that above a certain thickness the conductivity can decrease because of porosity [2].

Lewis [45] has reviewed some of the very extensive studies which have been made of the electrical resistance of thin films deposited by cathodic sputtering. The resistance properties of films prepared by this method will differ from those prepared by evaporation because the processing conditions are not the same. The high resistivity and negative temperature coefficient is usually found for thin films prepared by either method but the resistance changes after deposition can be much greater for evaporated films. Evaporated films can be condensed at very low pressures on to degassed supports and Appleyard [39] has shown the effects of substrate gas adsorption on film resistivity, whereas sputtering requires the presence of a gas at a relatively high pressure. Films deposited by evaporation can be formed rapidly but sputtered films usually take several minutes to form. The temperature rise in the substrate and film is also usually much greater for sputtering unless the work table is cooled.

The general tendency in sputtering is to produce a film which is more stable than evaporated films but coarser in structure, both in size and number of aggregates. The author has found that sputtered silver films are very often diffuse in appearance and only where the sputtering is rapid is it possible to produce specular films.

It is of interest to note that sputtered films are still employed on barrier-layer photo-cells of the selenium type, where the film thickness must be critically adjusted, depending on its conductivity and light transmission to obtain maximum power output. It

is understandable that the changes which take place in evaporated films after deposition would make such a critical adjustment difficult.

The author investigated the resistance changes of nichrome films evaporated on to glass and the graph (Fig. 5) shows some typical examples of the resistance variations measured. A protective silica film was deposited on to some of the resistance films before exposure to air and the results have been plotted (Fig. 6). During the deposition of the silica film radiation from the vapour source raised the temperature of the nichrome film and, consequently, in accordance with the negative temperature coefficient of the film, its resistance decreased, as in curve (*a*). Switching on a heater without depositing silica had a similar effect—see curve (*b*). Admission of air to the chamber produced only a negligible resistance rise but under the same conditions a similar film with no protective layer increased its resistance by 30% in 10 minutes. To complete the experiment, a second sample of an unprotected film showing a similar rise of resistance following the admission of air, was re-pumped and coated with silica. Thereafter, exposure to atmosphere did not increase its resistance. To determine the effect of the substrate on the resistance changes a silica film was condensed prior to the nichrome film and the results are graphically shown in Fig. 7.

Silica is a difficult material to evaporate and its substitution by magnesium fluoride was investigated. It was found that reasonable stability of the resistance could be achieved by applying magnesium fluoride with the substrate temperature in the region of 300°C. It was noted that if the resistor temperature exceeded this value the condensed MgF_2 disintegrated the nichrome film.

From these results it would appear that the conductivity of nichrome films is decreased on exposure to air by oxidation or possibly by an adsorption process. As expected, these changes are greatest for very thin films. Protecting the resistor surface from the atmosphere allows the observation of crystallisation. . The alteration in conductivity after deposition, due to crystallisation, can be prevented by raising the temperature of the substrate during evaporation.

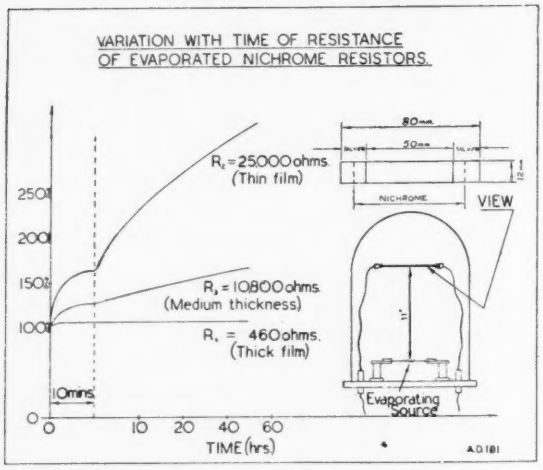

Fig. 5. Resistance changes in three films of different thickness after formation and exposure to the atmosphere.

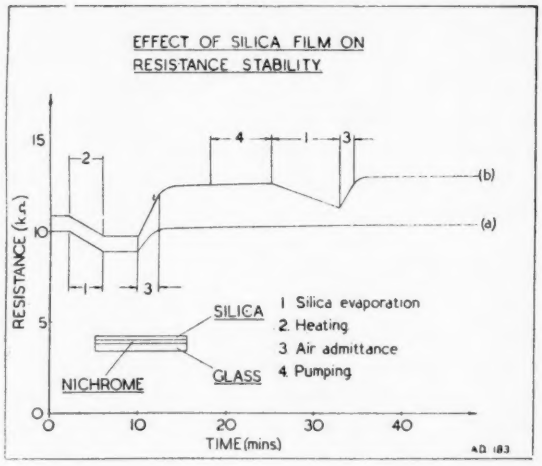

Fig. 6. Effect of sealing the metal film surface with a silica layer to prevent oxidation on exposure to atmosphere. Negative temperature coefficient of film is shown by decrease in resistance, when the film is heated *in vacuo*.

Vol. 1 1951

Effects of Residual Gases and Vapours

The pressure and nature of the residual gases present during evaporation have an important effect on both the structure and degree of contamination of the condensed substances. Increasing the pressure of the residual gas usually decreases the deposition rate and produces diffuse deposits. Degassing vapours, such as water, hydro-carbons, etc., may decompose on striking the evaporation source releasing active gases and evaporated metals may evolve gases during preliminary heating; all of which may, in some manner, react with the evaporated substance.

Stahl and Wagener[46 46a] studied the oxidation of a number of metals deposited on to the specimen surface inside an electron diffraction camera at a gas pressure of 10^{-4} mm.Hg. They observed in the case of Aluminium, Beryllium, Molybdenum, and Nickel only the metal oxide lattice up to a thickness of 0.05 micron and found that the metal lattice appeared at a thickness above 0.1 micron only after continuous evaporation, during which the chamber pressure was lowered by gettering. Further tests on slow evaporation to prevent gettering from lowering the pressure, produced in the case of magnesium, the oxide lattice even at one micron. The figures given should be treated with caution, since an opaque film of aluminium is approximately 0.05 microns and such intense oxidation has never been found at 10^{-4} mm.Hg pressure by the author.

From various tests Stahl and Wagener conclude that oxidation must take place either whilst the metal is in the vapour phase or immediately it is condensed. Other tests showed that a high substrate temperature, greater than 300°C., is normally required to oxidise a metal film after condensation, whereas their substrate temperature was never greater than 70°-100°C.

For the intense oxidation to occur by gas-vapour molecular collisions in the source-receiving surface space (= 1 cm. gap) the meanfree path of aluminium molecules in air would need to be of the order of one cm. at 10^{-4}mm. Hg, whereas in fact the mean free path is of the order of 160cm. The second possibility of oxidation immediately on reaching the substrate surface is likely since the condensed metal atoms will on some substrates retain their energy for an appreciable time and, if the deposition rate is very low, the chances of collision on the substrate surface between metal atoms and oxygen atoms will be greater than between metal atoms. The structure of films is dependent on the rate of formation and it may easily be that this would have some effect on the degree of oxidation of thin films.

If the results of Stahl and Wagener are treated qualitatively it would appear correct to say that rapid evaporation produces coatings showing a minimum of oxidation.

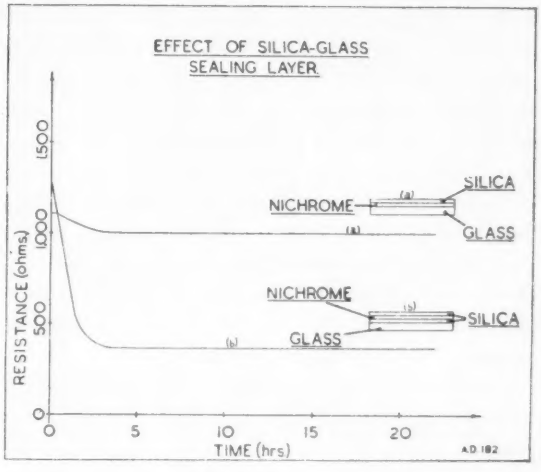

Fig. 7. Resistance changes for thick films (*a*) a film of low initial resistance, *i.e.*, a comparatively thick film showing a resistance fall when protected with silica. (*b*) demonstrating the remarkable influence of the substrate. The normal glass surface has been filmed with silica and thus the metal (nichrome) film is sandwiched between two silica layers: the resistance fall is even greater.

Crystalline Structure

The crystalline structure of thin films is influenced by the nature and temperature of the substrate, *i.e.*, a thin film usually tends to follow the orientation of crystalline substrates whereas on glass surfaces orientation can take place with one plane of the film crystals parallel to the substrate surface, the degree of orientation being determined by the substrate

temperature. Finch and Quarrell [47] have shown that for thin films of aluminium deposited on to a platinum substrate, the basal dimensions of the aluminium lattice is similar to that of platinum. Finch [48] states that such pseudomorphic lattice changes have only been observed, where the lattice constants and crystal structure of the deposit and substrate are similar.

Levinstein [49] has recently examined the structure of evaporated metal films by electron diffraction and electron microscopy. He found that both the size and orientation of the metal crystals can be correlated to the melting point of the metal from which the film is being formed. Films formed from high melting point metals consist of micro-crystals with no specific orientation whilst films made of low melting point metals produce large crystals orientated preferentially to the substrate. He also found that for mono-atomic vapour the velocity of the vapour molecules did not influence structure. This is at variance with a number of workers who have inferred that properties such as hardness, etc., of condensed films are largely dependent on this factor. Levinstein further showed that the crystal structure of the condensed film was indirectly determined by the rate of formation of the film because of its influence on grain size.

Sennett and Scott [50] have measured the optical constants of evaporated films and compared them with film structure. They found, in agreement with Levinstein, that the rate of formation of the film affected the structure and also the optical properties. In general, slow rates of formation produced a more aggregated structure and increased light absorption. They concluded that a high rate of deposition is required for the production of high quality reflection coatings. Andrade and Martindale [51] also observed the formation of an aggregated structure in sputtered silver films after prolonged heating and considered that many of the properties attributed to sputtered films are caused by heating of the film during deposition.

The rate of formation of the film is determined by the number of atoms striking the condensing surface in unit time and is proportional to the evaporation rate of the substance being vaporised. Since an increase in source temperature increases the vapour intensity and molecular velocity it is not surprising that a number of workers have been led to believe that the latter was a deciding influence in film structure.

Theory of Condensation

The deposition rate has another very important influence on the formation of the condensed film. In fact, it has been shown by a number of workers that if the intensity of the vapour beam is below a critical value, films cannot be formed at all. This has been explained by assuming that, if the substrate binding forces are weak, deposited atoms move over the substrate and eventually re-evaporate from the surface. Thus a deposit can only be formed if the number of atoms impinging on the surface is greater than the number re-evaporating. The chance of atoms colliding on the substrate is greater if the beam intensity is increased and thus atom pairs will be formed with a longer surface lifetime. The atom pairs then act as nuclei for condensation. The critical vapour intensity is, therefore, determined by the lifetime of a condensed atom on the substrate. Corresponding to the critical vapour intensity is a critical surface temperature, *i.e.*, for a given beam intensity there exists a maximum substrate temperature above which a film cannot be formed [52].

Usually, these phenomena only occur in the case of volatile substances, such as zinc and cadmium and where the forces between the substrate surface and condensed molecules are weak. Frazer [53] dealing with this subject states that such transition phenomena should not be expected to occur on metal substrates which have been degassed properly but only on metal surfaces contaminated by adsorbed gases or grease which reduce the surface forces of the substrate. In the case of thoroughly degassed metal substrates the vapour should condense at even the weakest intensities and the deposited layer should conform with the crystal lattice of the substrate.

Rhodin [54] has made a study of the crystal orientation of aluminium films on a number of crystalline substrates. He found that there was a

Vol. 1 1951

characteristic temperature for the substrate which produced maximum orientation and assumed that the condensed atoms require a minimum kinetic energy to allow them to move into the preferred positions. The binding energy between the condensed atoms and the crystalline surface was determined for a number of substrates from measurements made of the minimum vapour intensity required to ensure film formation, with the substrate kept at the temperature for which maximum orientation was known to occur. From the results obtained it was possible to show a relationship between degree of orientation and binding energy. For collodion and glass substrates Picard and Duffendack [55] found film aggregation was greatest for zinc and cadmium; they concluded that in the case of Aluminium, Copper and Gold, the surface temperature required for mobility to form aggregates was much higher.

Evaporated films are usually deposited on to degassed surfaces and, consequently, films can be formed with low rates of evaporation.

Various methods, which are all consistent with the theory outlined above, have been described to ensure the formation of zinc and cadmium films on glass, *e.g.* (*a*) the substrate surface temperature is lowered by cooling with liquid air [52] (*b*) the surface forces are increased by pre-coating with a metal such as silver or chromium [54] or, (*c*) the vapour source is constructed to produce high intensity vapour beams [52]. In the case of the latter method, the zinc and cadmium films produced are usually made up of large crystals, due to the high surface mobility of their atoms at ambient temperatures and also because of increased thickness which is difficult to control at high rates of evaporation.

CONCLUSIONS

From the many investigations reviewed four principal factors emerge as determining the structure and properties of films deposited *in vacuo* :—

(1) the nature and temperature of the substrate obtaining during and after deposition.

(2) the evaporation rate and mass of substance condensed per unit area.

(3) the pressure and nature of residual gases present during evaporation.

(4) the degree of adsorption and oxidation occurring in the films subsequent to its formation.

It should be apparent that considerable attention is being devoted to the development of apparatus for the preparation of thin films and to research into their formation, structure and properties. As a result, it is safe to say that the application of vacuum coating for scientific and industrial purposes will continue to increase.

I should like to acknowledge permission and encouragement to publish this account given me by Mr. A. S. D. Barrett, Technical Director of W Edwards & Co. (London) Ltd., and the suggestions made by colleagues who kindly checked the text. Acknowledgments are also made to the Medical Research Council for the loan of Fig. 3.

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Vol. 1 1951

LETTERS TO THE EDITOR

Vapour Pressure Measurements of Bromine

Sommaire

L'AUTEUR DECRIT des expériences sur la mesure de la tension de vapeur du brome. Tout d'abord il s'était servi d'un manomètre à huile de silicone. Mais afin de rendre les mesures plus précises, les expériences furent répétées ensuite avec une jauge du type Pirani à fil de platine. Pour augmenter la précision encore plus on se propose de faire usage d'un manomètre de viscosité à fil de quartz. Les résultats de ces dernières expériences seront publiés plus tard.

IN CONNECTION with investigations on electric discharges in gases it became necessary to measure pressures of bromine vapour of the order of 0.1 mm. Hg. The vapour was introduced into a vacuum system from a small side tube provided with a tap. In order to exhaust the air from this tube in the presence of liquid bromine it was placed in a Dewar flask containing liquid air. This caused the bromine to solidify (m.p. = −7.3°C) and its vapour pressure to be reduced to a negligible value. The air was then removed using a two-stage mechanical rotary pump. The actual vacuum achieved was 10^{-3} mm.Hg.

To measure the pressure of the bromine vapour subsequently established in the system, a silicone oil manometer was used in the first instance. Figure (1) gives a diagrammatic sketch of the manometer. Taps T_1 and T_2 were opened to connect the manometer with the evacuated system. Then the oil was heated for 30 minutes to facilitate liberation and evacuation of the air dissolved in the oil. On closing tap T_1 the aforementioned vacuum of 10^{-3} mm.Hg was maintained in the bulb B, whilst opening tap T_2 allowed the bromine vapour to be introduced into the manometer and measurement of its pressure by the depression of the oil level L_1 against L_2. The use of a travelling microscope ensured an accuracy of readings within 0.05 mm. Considering that silicone 702 oil has a specific gravity of 1.07 the pressure of bromine could be read with an accuracy of 0.05 x 1.07/13.6 = 0.004 mm.Hg. The major difficulty in this experiment was the fact that the bromine vapour dissolved in the oil and thus entered into the evacuated bulb B of the manometer. The vapours in bulb B exercised a counter pressure on the level L_2. In order to counteract this effect and prevent an excessive pressure rise during the experiment, the bulb B was made of considerable volume. Nevertheless this difficulty reduced the accuracy of measurement to a margin of 0.02 mm.Hg., i.e. an error of 20% in measuring a pressure of 0.1 mm.Hg.

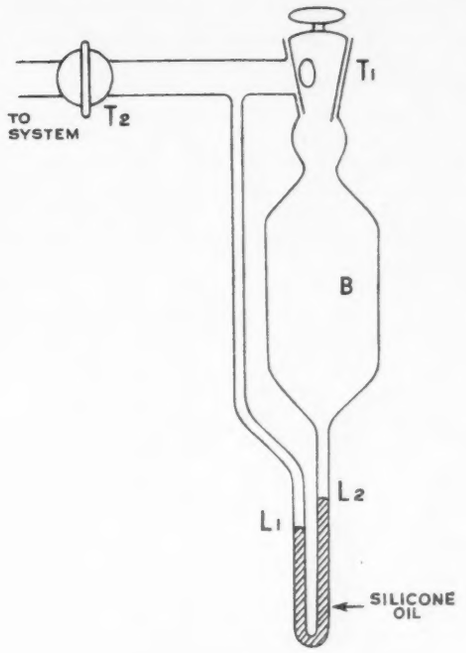

Fig. 1.—Oil Manometer

In an endeavour to improve on this, a Pirani gauge with a platinum filament was used. The envelope of this gauge was of borosilicate glass which is much less subject to attack by bromine than the sodalime and lead glasses normally employed. Using Knudsen's formula for the conductivity of free molecules[1] and proceeding on the assumption that the ratio of the specific heats (at constant pressure and constant volume) as well as the accommodation coefficient is constant, the calibration of a Pirani gauge is approximately proportional to $1/\sqrt{M}$, where M is the molecular weight of the gas in the gauge. Thus the reading from bromine vapour (M=160) should be $\sqrt{\frac{28}{160}}=0.42$ of that for nitrogen (M=28). An experimentally obtained calibration curve of the Pirani gauge for nitrogen gas compared with the

curve obtained for bromine vapour, using the silicone oil manometer as an absolute gauge, showed a ratio of sensitivities of the order expected, though the ratio of 0.42 was not uniform over the range from 0.01 to 1.0 mm.Hg because of departure from true free molecule conductivity at the higher pressures. Nevertheless, the calibration obtained was reproducible and considered satisfactory.

It is now proposed to use a quartz fibre viscosity manometer. The calibration date of this gauge are known for hydrogen ($M = 2$) and xenon ($M = 131$) in the pressure range required. The viscosity of gas at low pressures is dependent on $\sqrt{M}$, thus the time for the half amplitude to be reached in observing the oscillation of the quartz fibre should be appreciably less for bromine than for xenon, i.e. appreciably less less than 5.3 seconds at a pressure of 0.1 mm.Hg. This manometer, operated at room temperature and with a small volume bulb, should ensure even greater accuracy of measurement in connection with the experiment discussed.

More information will be made available after completion of these experiments.

J. Yarwood.
D. H. Le Croissette.

The Polytechnic,
Regent Street,
London, W.1.
1st May, 1950.

[1] DUSHMAN, S., *Scientific Foundations of Vacuum Technique.* (John Wiley & Sons Inc. New York, 1949), 51.

Density Control in the Manufacture of Rhodium Filters

Sommaire

ON SE HEURTE à trois principales difficultés dans la fabriquation des filtres en rhodium à faible densité optique : 1) La valeur de la densité du dépôt continue à augmenter pendant quelque temps aprés la fin d'une évaporation, pour arriver à une valeur stable. (2) Le brunissage fait changer cette valeur stable. (3) La faible densité aux bords de la couche donne au filtre une densité moyenne plus faible, tout particulièrement dans le cas des filtres à petites dimensions.

RHODIUM is a very suitable material for the production by means of evaporation in vacuo of high quality optical filters. Its special features are these:—

(1) Its transmission changes only very slowly with wavelength and can be taken as neutral over fairly wide spectrum ranges.

(2) Its durability is excellent.

(3) Owing to the comparatively slow rate of evaporation, an accurate density control is possible.

There are, however, a few not quite obvious difficulties which become noticeable only if one tries to produce low density steps of the order of, say, 0.2 - 0.3, with an accuracy of 5% or better. If one does not take special precautions, surprisingly large errors are sometimes obtained. As this may be of interest to many workers, it was thought to be worth while mentioning a few points concerning this problem.

During manufacture the density is measured in the usual way, i.e. a beam of light obtained from a constant source, enters the bell jar through a window, passes through the sample to be coated, and leaves the bell jar through another window. The light then falls onto a photocell and the photocurrent is measured by a galvanometer. During evaporation, the entrance window is covered from inside by a shutter, while the exit window is protected by the sample and, if necessary, by an additional baffle. If the area to be coated is very small and limited by a mask the filament of the light source has to be focused onto the sample. Before evaporation is started, the shutter is opened, and the galvanometer deflection is set to full scale corresponding to 100% by adjusting the sensitivity control. Then the shutter is closed and evaporation is started. After a while, evaporation is stopped, the shutter is opened, and a reading is taken. If the transmission is still too high, the procedure is continued until the desired density is reached.

It is at this point that a peculiar trouble is frequently experienced—well after stopping evaporation, the density of the rhodium film still rises. Suppose one has stopped evaporation when a density 0.20 (63% transmission) was reached. If one measures the density again two minutes later, without any more evaporation having been done, one may find a density 0.23 (59% transmission) and after another two minutes possibly 0.24 (57.5 transmission). The rate of increase gradually slows down, and eventually the density reaches a final value, but in some cases one may have to wait up to five minutes for it to settle. In general, one can say that the effect increases with greater film thickness deposited in one continuous run and with higher rate of evaporation. In order to produce a given density, therefore, one

Vol. 1 195

has to stop evaporation well before this value is reached, and wait, and then add a little and again, and so on until the required density is reached.

Another cause for obtaining wrong densities is burnishing. Rhodium films are usually burnished immediately after being taken out of the vacuum chamber. This process increases the density. The effect is small but sufficient to upset high precision values. This burnishing effect is also partly responsible for errors in the case when the burnished rhodium films are put back into the vacuum chamber for a second or third coating.

It is well-known that double or triple coatings are the only reliable way of producing pin-hole free films. The density of filters of this type has to be made up by the densities of the two or three coatings. These densities are always measured before burnishing while the filters are still in the vacuum chamber. It is objectionable to measure them after burnishing outside the vacuum chamber because of the serious risk of surface contamination. Conditions become even more complicated when the rhodium films are exposed to another glow discharge before the next coat is deposited. The effect of the discharge on the rhodium film is a small drop in density, but whether this drop over-compensates, eliminates, or under-compensates the density increase due to burnishing, depends on many practical conditions.

Finally, a rather more trivial cause for density errors might be worth while mentioning. It occurs, when the filter area to be coated is limited by a mask and smaller than the patch of light used for measuring, so that part of the light falls on to the mask. One does not always realise that the density of a film is not uniform right up to the edge of the mask, but only up to a few hundredths of a millimetre distance from that edge depending on the geometry of mask and vapour source. Within that distance the density drops in a steep gradient down to zero. Although now the percentage of incident light passing through this narrow border area is very small one must not forget that the mean density of this narrow area is very small too, so that serious errors may be caused if the filter densities are of the order of 1.0 or higher.

It is impossible to give general correction figures for the errors mentioned, as they largely depend on individual conditions. If however the existence of these sources of error has been recognised it will be possible to find by trial and error modified density figures, which will give the correct density on the finished filter, providing a standard set up, and standard discharge, and evaporation routine is being adhered to.

W. Zehden.

Research Department,
Hilger & Watts Ltd.,
London,
England.
1st September, 1950.

BOOK REVIEWS

Le Vide et Ses Applications. (Vacuum and its Applications). By LOUIS DUNOYER. (Presses Universitaires de France, Paris, 1950). Pp. 112. Price 3s. 3d.

THIS moderately priced, paper-backed volume, written by a physicist whose name is familiar to all those interested in vacuum technique, is recommended not only as a good introduction to a subject of ever-growing importance, but also as a monograph which can be read with profit by the experienced technologist since it evinces much of that insight which is only gained by an author who has spent many years considering the intricacies of his subject.

A brief, clearly written account of the relevant aspects of the kinetic theory of gases, including the development of the important formulae for the flow of gases through tubes at low and intermediate pressures, is followed by straightforward details of the various vacuum pumps of the mechanical-rotary, diffusion-condensation and molecular types; it is interesting to note that the last of these seems to find much more favour in France than elsewhere. Two useful methods of measuring pump speeds are then considered together with the practical aspects of the pump characteristics which they obtain. A short account follows of the more important calculations necessary to set up vacuum pumps to give optimum performances.

Chapter III deals with the principles of working of the most useful types of vacuum gauge. It is worth noting here the details of the attempts by the French to standardise the construction and use of the ionisation gauge so that different laboratories and workshops can obtain comparable results.

The last two chapters are headed "Le Vide dans des Appareils Scellés", and "Quelques Applications du Vide dans des Appareils Non-Scellés". The first deals with the production of sealed-off tubes where baking, vacuum-stoving and gettering are of concern, whilst the second considers the so-called kinetic, or demountable vacuum systems such as those used for nuclear physics research, evaporation of metals *in vacuo*, vacuum drying and the production of penicillin and dehydrated orange juice.

The book suffers greatly from lack of illustrations: there are only twenty-six simple line drawings. Moreover, the later sections dealing with applications of the art contain no diagrams at all. Consequently, the industrial uses of vacuum are hardly likely to be adequately understood by the uninitiated. Again, in a small volume, it is inevitable that several topics are left undiscussed, but it is a pity that nothing is said about leak finding. Nevertheless, it would be difficult to obtain in England today any book on technology for only three shillings which is so competently written as this attractive little volume.

J. Yarwood.

Vol. 1 1951

VACUUM
CLASSIFIED ABSTRACTS

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

Vol.
1
1951

Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

(I) General Science and Engineering

10 — VACUUM ENGINEERING — GENERAL
11 —
12 —
13 — ELECTRICAL SCIENCES
14 — MECHANICAL SCIENCES
15 — PHYSICS
16 — CHEMISTRY
17 —
18 —
19 — SCIENCES (other than the above)

(II) Vacuum Apparatus and Auxiliaries

20 — PUMPS
21 — GAUGES
22 — MEASURING PLANT — VACUUM
23 — INSTRUMENTATION
24 —
25 —
26 — MATERIALS
27 —
28 — APPARATUS COMPONENTS
29 — MISCELLANEOUS APPARATUS

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
32 —
33 — IMPREGNATION
34 — FUMIGATION
35 —
36 — DRYING
37 — METALLURGICAL PROCESSES
38 — DISTILLATION
39 — MISCELLANEOUS PROCESSES

(IV) Special Subsidiary Subjects

40 — GASES & VAPOURS
41 — FREEZE DRYING
42 —
43 —
44 —
45 — ELECTRONICS
46 —
47 — GLASS PROCESSING
48 —
49 —

Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) A Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract *e.g.*, 20/**I** as distinct from 20/**II** etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

Cumulative Subject Index

Subject — Abstract No.

I — General Science and Engineering — I

10 — VACUUM ENGINEERING (GENERAL) — 10

Distillation 1/I; 2/I;
Electrolytic Polishing 4/I;
Gas Evolution 6/I;
Gas Supply to Vacuum Systems 5/I;
Pumps 2/I; 3/I;

13 — ELECTRICAL SCIENCE — 13

Insulating Means (Vacuum as an —) 7/I;
Resistors 8/I;

15 — PHYSICS — 15

Crystals (Metallic —) 12/I; 13/I;
Evaporation Rates 11/I;
Organic Molecules (Measurement of —) 14/I;
Photo-Conductivity 10/I;
Radiography 9/I;

16 — CHEMISTRY — 16

Antibiotics 19/I;
Blood Plasma 18/I;
Fats (Animal —) 16/I;
Fermentation 17/I;
Food Preservation 15/I;
Oils (Vegetable —) 16/I;

II — Vacuum Apparatus and Auxiliaries — II

20 — PUMPS — 20

Charged Particle Accelerators (Vacuum Plant for —) 3/II;
Circulating Pumps 2/II;
Piston Pumps 1/II;
Toepler Pumps 2/II;

21 — GAUGES — 21

Bellows Manometer 9/II;
Ionization Gauge 4/II; 5/II; 12/II; 13/II;
Knudsen Gauge 6/II;
McLeod Gauge 7/II; 8/II;
Mercury-Column Manometer 14/II;
Philips-Type Gauge 15/II; 11/II;
Pirani-Type Gauge 4/II; 10/II;

22 — MEASURING PLANT (VACUUM) — 22

Electron Diffraction 18/II;
Electron Microscope 17/II; 19/II; 32/II;
Mass Spectrometer 16/II; 27/II; 28/II; 29/II; 31/II;
Specimen Preparation 20/II; 21/II; 22/II; 23/II; 24/II; 25/II; 30/II;
Telescope (Electronic —) 26/II;

Vol. 1 1951

Cumulative Subject Index
Contd

Subject	Abstract No.
26 — MATERIALS — 26	
Porcelain	33/II;
Refractories	34/II;
28 — APPARATUS COMPONENTS — 28	
Gaskets	38/II;
Motors (Electrical —)	37/II;
Valves (Mechanical —)	35/II; 36/II;
29 — MISCELLANEOUS APPARATUS — 29	
Gases (Storage of Liquefied —)	43/II;
Leak Detector	41/II; 42/II;
Pressure Control	39/II; 40/II;
III — Vacuum Processing Techniques — III	
30 — EVAPORATION — 30	
Alkaline Earth Layers	17/III;
Aluminizing of T.V. Tubes	7/III;
Crystals (Growing of —)	5/III;
Etching	6/III;
Photo-sensitive Layers	16/III;
Physics of Evaporation	20/III;
Plastics (Deposition of Metal on —)	1/III; 4/III;
Precious Metals (Deposition of —)	15/III; 18/III;
Reflection-Reducing Films	3/III;
Replica Techniques (Various —)	11/III;
Resistors (Manufacture of —)	19/III;
Shadow Casting	8/III; 9/III; 10/III; 12/III; 13/III; 21/III;
Structure of Films	14/III;
Thin Films — General	2/III;
31 — CATHODIC SPUTTERING — 31	
Etching	23/III;
Thin Films — General	22/III;
36 — DRYING — 36	
Antibiotics	25/III;
Strip Material	24/III;
37 — METALLURGICAL PROCESSES — 37	
Carbon Determination	29/III;
Distillation of Metals	34/III;
Furnaces & Ovens	28/III; 31/III; 32/III;
Fusion Methods (Vacuum —)	30/III; 31/III;
Lost Wax Process	26/III;
Oxygen Determination	30/III;
Preparation of Metals	33/III;
' Rare ' Metals (Processing of —)	27/III; 35/III;

Cumulative Subject Index
Contd.

Subject	Abstract No.
38 — DISTILLATION — 38	
Developments (General —)	36/III;
Fat Extraction	40/III;
Fats (Distillation of —)	39/III;
Fractionating	37/III; 39/III;
Molecular	38/III; 41/III;
Safety Devices	42/III;
39 — MISCELLANEOUS PROCESSES — 39	
De-Odorization	44/III;
Food Processing	45/III; 46/III; 47/III; 48/III;
Vitreous Enamelling	43/III;
IV — Special Subsidiary Subjects — IV	
40 — GASES AND VAPOURS — 40	
Analysis of Gases	1/IV; 6/IV;
Blending (See Filling, Mixing & Blending)	
Circulating of Gases	5/IV;
Dew-Point-Bubble Point Apparatus	3/IV;
Evolution of Gases	8/IV;
Filling, Mixing & Blending	4/IV;
Liquefied Gases (Storage of —)	7/IV;
Organic Vapours (Determination of —)	2/IV;
41 — FREEZE-DRYING — 41	
Blood	9/IV;
Fermentation	12/IV;
Food	13/IV;
Tissue	10/IV; 11/IV;
45 — ELECTRONICS — 45	
Charged-Particle Accelerators	16/IV;
'Clean-up' — Phenomena	20/IV;
Oxide-Coated Cathode	14/IV; 15/IV;
Thermionic Emission	17/IV; 15/IV;
Valves (Electronic —)	18/IV; 19/IV;
47 — GLASS PROCESSING — 47	
Glass-To-Metal Seals	21/IV; 22/IV;

Vol. 1 1951

I — General Science and Engineering — I

Abstract No. and References

10 — VACUUM ENGINEERING (GENERAL) — 10

High Vacuum Distillation
See Abstract No.: 36/III
1/I

The Manufacture of Mononitrotoluene
See Abstract No.: 37/III
2/I

How To Figure Reciprocating Vacuum Pumps
See Abstract No.: 1/II
3/I

Electrolytic Polishing and Vacuum Engineering
4/I

France. Electrolytic polishing is discussed as it has great merits in connection with the finishing of components used in the assembly of electronic tubes. The two principal advantages of this method are: (1) The elimination of some of the adsorbed gases and (2) The suppression of the cold emission of tubes designed to run at a very high tension.

Sommaire: L'auteur discute les principaux avantages du polissage électrolytique, tout spécialement du point de vue des ingénieurs du vide.

Article by P. Nineuil *Le Vide*, 5, May 1950, 807-813

A Method of Supplying Low Pressure Gases to a Vacuum System
5/I

United States. In some vacuum processes it is necessary to feed a quantity of gas into the system at certain stages of the procedure. As the gas to be admitted may be subject to reactions or absorption phenomena in the system the supply of gas cannot be easily defined and the process of feeding is not simply one of filling and sealing at any given pressure. Waste of gas may thus be incurred—an important factor in the case of high purity gases. A method is described here which is used in connection with the processing of photo-conductive cells. It essentially consists of a manifold placed between fore-pump and diffusion pump with a line by-passing the diffusion pump. Two mercury cut-offs are provided, one in the by-pass line and one in front of the fore-pump. After pumping down to the desired level of pressure the fore-pump is cut off and the gas introduced into the manifold. The mercury cut-off in the by-pass line is opened and the gas allowed to circulate. After adjustment of the pressure, the cut-off in front of the fore-pump is closed and thus the system isolated from all stopcocks. The pressure in the manifold can now be changed rapidly from one value to another simply by operating the mercury cut-off in the by-pass line.

Sommaire: On décrit un système pour alimenter une enceinte à vide avec un gaz.

Note by R. H. McFee *Rev. Sci. Instrum.* 21, Jan. 1950, 100-101

On Certain Phenomena Concerning the Release of Gases Observed in the Production of Low-Voltage Incandescent Lamps
6/I

France. Whilst pumping incandescent lamps of the 2-volt type the author observed that the lighting-up of the filament causes liberation of gas which is the more rapid and plentiful, the longer the interval has been between the final de-gassing process and the first lighting-up of the lamp. The amount of the gas liberated is very small but it was possible to arrive at a close assessment with the help of the micro-manometer described by the author elsewhere. The investigation led to the conclusion that the release of gas is due to a progressive formation—even at a very high vacuum—of adsorbed layers on the surface of the bulbs in spite of thorough cleaning operations carried out previously. It is assumed that these depositions originate from the diffusion of the top layers of gas absorbed by the walls of the pumping lines in the direction of the glass bulbs. A theory is advanced supporting this hypothesis. From a practical point of view this investigation seems to prove that sealing operations should not be unduly delayed. Otherwise, in the absence of a getter, such depositions will accumulate and volatilise later.

Sommaire: Sur certains phénomènes de dégagement gazeux observés pendant le pompage de lampes à incandescence à basse tension.

Article by L. Dunoyer *Le Vide*, 5, May 1950, 793-806

13 — ELECTRICAL SCIENCE — 13

Insulation for High Voltage Apparatus
7/I

United States. The past and future of transformer design is discussed. Some engineers, it is stated, believe that developments may lead to the provision of a vacuum as a means for insulating high voltage apparatus. This would overcome the danger of breakdowns due to ionization and generally simplify the problem of electrical insulation. On the other hand, the task of heat disposal would be made difficult unless the amount of heat originated at the source could be reduced by the application of low-loss cores and low-loss conducting materials.

Sommaire: On envisage l'usage du vide comme isolateur dans les appareils à haute tension.

Note by F. L. Snyder *Westinghouse Engr.* 10, Jan. 1950, 61

I — General Science and Engineering — I
Contd.

Abstract No. and References

Experimental High Megohm Resistors 8/I

United States. The consistency and accuracy of measurements with analytical mass spectrometers could only be ensured by overcoming a number of electronic problems. One of them is the provision of high-value resistors in the electrometer circuit. Several types of resistors with a range of about 4×10^{10} Ω have been developed. The type considered to be the most successful in performance consists of a glass tube with two tungsten wires sealed in at either end. From these wires metal is evaporated (*in vacuo*) and deposited on the glass walls connecting the two resistor leads. Care must be taken to prevent the tungsten from oxidising otherwise the resistance value of the film will be reduced considerably.

Sommaire : On décrit des résistances à valeur élevée produites par évaporation dans le vide.

Note by K. E. Burmaster *Nucleonics*, 7, July 1950, 79

15 — PHYSICS — 15

New Techniques in Autoradiography 9/I

See Abstract No. : 10/IV

The Photo-Conductivity of Bismuth Sulphide and Bismuth Telluride 10/I

See Abstract No. : 16/III

Evaporation and Outgassing in an Inert Atmosphere 11/I

United States. Outgassing and evaporation rates in a vacuum proper depend on the respective reflection or accommodation coefficients. But in the presence of an inert atmosphere these rates are determined by a diffusion process. Here, a great number of evaporated molecules are reflected by the gas and return to the evaporation surface, where they condense. Experiments have been conducted to determine these conditions and are described in this article. Two samples were used : (*a*) Antimony steel shims 0.005 in. thick and (*b*) Antimony films deposited on glass slides by an evaporation process. The density of the films was 16μg/cm^2. These samples were fixed to a Gulbransen micro-balance and were suspended in a tube which could be heated by external means. Helium was used as an inert gas. It was found that in the presence of an inert gas at a pressure of 0.1 atmosphere the outgassing rate was reduced by a factor of 7 and the evaporation rate was reduced by a factor of 100-1,000. Thus an inert gas atmosphere effects the reduction of the evaporation rate down to a very small fraction of the outgassing rate.

Sommaire : On étudie les effets d'un gaz inactif sur l'évaporation et le taux de dégazage de l'antimoine.

Article by R. L. Longini *J. Appl. Phys.*, 21, Feb. 1950, 81-3

The Thermal Etching of Single Crystals of Cadmium 12/I

United Kingdom. In connection with an investigation on the effects of surface conditions on the mechanical behaviour of single crystals of cadmium, in the form of 1 mm. diameter wires, it seemed desirable to try to obtain the cleanest possible surface. Mechanical polishing could not be used because it tends to deform the crystal structure of soft wires and electrolytic polishing has a marked effect on the mechanical properties of the wire which are not fully clarified yet. It was decided therefore to remove the surface layers of the wire by evaporation *in vacuo*. The actual production of the crystal to be investigated was carried out by the Andrade-Roscoe travelling furnace method with the wire being placed in a pyrex tube which is evacuated to a pressure of approximately 3×10^{-4}mm.Hg and cleaned beforehand *in vacuo* at a temperature of 400°C. Two methods are used for the evaporation : (*a*) Heating the wire by passing an electric current through it and (*b*) by the travelling furnace method. The second method is in fact a combination of the actual crystal formation and etching. The results of the investigation are given as follows :—When the basal plane makes a small angle with the axis of the wire, there is a characteristic formation of hexagonal pits, the bottoms of which are mirror-like basal planes; when the basal planes make larger angles, characteristic elliptical traces appear reminiscent of those formed by mechanical extension. The thermal etching reveals, by the formation of steps, thin laminae existing in the unstrained wires, the thickness of which (about 1μ) is the same as the spacing of the glide planes revealed by mechanical extension. The etched figures in general indicate that the substructure revealed by straining is already present in the unstrained crystal. The secondary pyramidal faces revealed agree with the calculations of Stranski, Kaischew and Krantanov.

It appears that the method of thermal etching has considerable potentialities in connection with investigation work on the secondary structure of single crystals.

Sommaire : On discute le ' dérochage ' thermique et la structure des crystaux de cadmium.

Article by E. N. Da C. Andrade and R. F. Y. Randall *Proc. Phys. Soc.*, 63, B, 1.3.1950, 198-207

On the Growth of Metallic Crystals from the Vapour Phase 13/I

See Abstract No. : 5/III

I — General Science and Engineering — I
Contd.

Abstract No. and References

Direct Optical Measurements of the Length of Organic Molecules — 14/I

United Kingdom. A procedure is described of measuring the thickness of mono-molecular layers of adsorbed organic materials based on optical techniques. In the particular experiment muscovite mica was used as a reference material, one facet of which is partly covered with a mono - molecular layer of stearic acid ($C_{17}H_{35}COOH$). Silver was evaporated onto both sides of the specimen until the reflection coefficients were greater than 90%. Monochromatic light of a low pressure mercury arc lamp was used and the specimen tilted to 4° from the normal so that the transmitted intensity for the facet was approximately one half of the maximum obtainable. In this way the uniformity of an adsorbed mono-molecular layer can be examined qualitatively. Finally details are given how to determine quantitatively its thickness at the same time.

Sommaire : L'auteur donne une mèthode pour dèterminer l'épaisseur d'une couche mono-moléculaire de matière organique adsorbée.

Article by J. S. Courtney-Pratt *Nature*, 165, 4.3.1950, 346-8

16 — CHEMISTRY — 16

Chemistry in Food Preservation — 15/I

United Kingdom. Research work on and processes connected with food preservation are reviewed. Drying and powderisation of eggs is mentioned, chilling of meat, the production of milk powder, spray drying and finally the potential advantages of evaporating water from frozen milk under a very high vacuum—a process not yet commercially established.

Sommaire : Une revue des recherches et des procèdés concernants la conservation des aliments.

Article by L. H. Lampitt *Times Science Suppl.* August 1950, 6-8

De-Odorization of Vegetable Oils — 16/I

See Abstract No. : 44/III

Technical Developments of Fermentation Processes — 17/I

See Abstract No. : 12/IV

Fractionation of Blood Plasma — 18/I

See Abstract No. : 9/IV

Antibiotics Development — 19/I

See Abstract No. : 25/III

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1
951

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

How to Figure Reciprocating Vacuum Pumps 1/II

United States. Data in graphical and tabulated form designed to assist the chemical engineer in calculations for the assessment of reciprocating vacuum pump systems. Examples are given how to calculate :—(1) The time required to evacuate a receiver of given capacity : (2) The power required for the drive of the pump and (3) the respective dimensions of the piping.

Sommaire : Ceci contient des données pour aider les ingénieurs chimistes dans leurs calculs sur les systèmes du vide faisant usage des pompes a piston.

Article by T. J. Hicks *Chem. Engng.* **57**, June 1950, 108-110

Glass Circulating Pump 2/II

United States. An arrangement is described for circulating gases which consist of two automatic Toepler pumps connected in parallel. This type of pump operates by positive displacement of circulating gas, therefore it can be used to circulate through restrictions such as stopcock bores and at sub-atmospheric pressures.

Continuous flow is effected by working the pumps one half cycle apart. The pumping action is controlled by an electrical circuit which actuates solenoid valves applying vacuum to the pump when energised and air pressure when de-energised.

Sommaire : On décrit un assemblage de deux pompes Toepler pour la circulation des gaz.

Note by F. C. Johnson and J. A. Glober *Analyt. Chem.* **22**, Jan. 1950, 204

Design Study for a 3-Bev Proton Accelerator 3/II

See Abstract No. : 16/III

21 — GAUGES — 21

The Pirani Effect in a Thermionic Filament as a Means of Measuring Low Pressures 4/II

United Kingdom. An ionisation gauge was used for the experiments consisting of a straight pure tungsten filament of .06 mm. diameter surrounded by a spiral wire grid and a co-axial cylindrical anode. The normal filament voltage and current was 3V and 1A respectively. The grid was earthed and the anode voltage kept at 165. The alternating voltage applied to the filament produced an emission current of 90 μA and could be varied by means of a Variac transformer. Three calibrations were carried out, two in air and one in hydrogen, and the Pirani cooling effect was investigated down to pressures of the order of 10^{-6}mm Hg. To maintain the emission current of 90μA in the pressure range of 10^{-1} mm. to 10^{-6} mm.Hg the heater voltages had to be changed by an amount large enough to be indicated by an ordinary pointer instrument. It is suggested therefore, that a low pressure gauge employing this effect could be used to cover both, the range normally covered by a Pirani gauge and that covered by an ionisation gauge.

Sommaire : Les auteurs décrivent des expériences sur l'éffet Pirani dans un fil de tungstène pour la mesure des basses pressions.

Article by L. Spiers and W. P. Jolly *Brit. J. Appl. Phys.* **1**, May 1950, 132-3

Vacuum Factor of the Oxide-Cathode Valve 5/II

See Abstract No. : 14/IV

Improvements in Measuring Devices Dependent on Radiometer Action 6/II

United Kingdom. A gauge is described of the Knudsen type in which the displaceable vane takes the form of a membrane, the movement of which relative to a fixed plate is measured in terms of a capacity change. This may be converted to a frequency change by making it part of a tuned circuit so that by heterodyning the pressure changes are indicated in the form of electrical signals.

Sommaire : Ce brevêt protège le projet d'une jauge radiometrique.

W. Steckelmacher and W. Edwards & Co. (London), Ltd. *Brit. Pat.* 630,885

A Rotary McLeod Gauge 7/II

United States. A rotary type is described made completely of glass with the exception of the '0'-ring vacuum seal. It consists essentially of 3 sections, the head of the gauge containing the open and closed capillaries, the coil or helix, and the end section which comprises the reservoir, cut-off and rotary seal. The amount of mercury used does not exceed 1 kg. Fitted with a 0.8 mm. capillary, the gauge constant is 1.4×10^{-6}mm.$^{-1}$. The gauge covers the pressure range of 2×10^{-6} to 2×10^{-2}mm.Hg.

Sommaire : Description d'une nouveau type rotatif de la jauge McLeod.

Article by M. Axelbank *Rev. Sci. Instrum.* **21**, June 1950, 511-513

Vol. 1 1951

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

A McLeod Gauge of Multiple Compression

8/II

Holland. The author describes a new manometer of the McLeod type, the sensitivity of which is being raised by applying the principle of multiple compression. Ordinarily only one fraction of gas is compressed in the measuring capillary. In this case, however, several fractions are being compressed, one after the other. This is facilitated by a small valve operating automatically. Pressure readings are taken in the usual manner after the last compression has been effected.

Sommaire : On décrit une jauge de McLeod à compression multiple.

Article by J. Groszkowski *Le Vide*, 4, July-Sept. 49, 668-672

The Telerecording of Thickness, Pressure Flow and Other Physical Quantities Using a Simple Electromagnetic Circuit

9/II

United Kingdom. A simple method is described of recording changes in induction in an electro-magnetic circuit and the adaptation of this method in connection with the design of a variety of industrial measuring instruments. Details of one of them, a bellows manometer, are given. This instrument indicates very small pressures and pressure differences of less than ± 0.01 mm.Hg. A full scale deflection on the instrument corresponds to .6 mm.Hg pressure difference, and a movement of the bellows of approximately 1×10^{-2} inchs per mm.Hg. No glands are used, and with the exception of the bellows there are no moving parts. A photograph of the instrument, circuit diagrams and calibration curves are given.

Sommaire : On décrit un nouveau circuit electro-magnetique employé dans un manomètre à soufflet, qui pourrait servir à des mesures de la pression et de la conductibilité thermique des gaz.

Article by W. J. Clark, *Trans. Soc. Instrum. Technol.*, 2, March 1950, 18-45

A New Pirani-Type Vacuum Gauge

10/II

Spain. Where Pirani gauges are used in connection with high vacuum distillation units, gauge stability in ' dirty ' atmospheres and gauge sensitivity to line voltage fluctuations present a serious problem. As to the former, the zero point shift, mentioned by Hickman first, was confirmed in experiments conducted by the authors. The gauge shows a tendency to read pressures higher than those actually existing in the system and this is explained by assuming that oil vapours disintegrate when in contact with the hot wire, producing a carbon deposit. Consequently, the emissive power of the filament increases and the resistance decreases. In an effort to overcome this trouble the authors covered the gauge filament with a fine glass capillary and worked the gauge at temperatures as low as possible. In addition, all metal parts of the gauge in contact with the atmosphere in the system were covered with glass to prohibit reactions with corrosive gases. These measures resulted in a very good zero point stability. No shift was noticed over a period of two years.

To reduce the sensitivity to line voltage fluctuations a current-regulating tube was inserted and connected in series with the bridge. Details of the circuit are given. The regulation afforded by these means is not perfect but has improved conditions.

Sommaire : Les auteurs font un rapport sur les measures à prendre pour rendre la jauge de Pirani stable dans une atmosphère ' sale ', comme par example dans lec procédés de distillation sous vide.

Article by E. Blasco and L. Miranda *Rev. Sci. Instrum.* 21, May 1950, 494-495

Construction and Application of a New Design of the Philips-Type Vacuum Gauge

11/II

Holland. The pressure range covered by the known type of Philips vacuum gauge extends from 2×10^{-3} to 2×10^{-5} mm.Hg. In order to produce a vacuum gauge based on the same principle but applicable to a range of pressures lower than the above, the conventional electrode system has been replaced by a design consisting of two parallel round metal discs as cathode, and an anode in the form of a cylindrical jacket.

To keep the distance between the pole shoes of the permanent magnet surrounding the glass tube as short as possible, the tube has been pinched at that point. These changes raise the sensitivity of the instrument by a factor of 10. The modified gauge covers a range of 10^{-4} to 10^{-6}mm. Hg and can probably be used for pressures below 10^{-7}mm.Hg.

Sommaire : Un nouveau modèle de la jauge de Philips permettant son usage à des pression même au-dessous de 10^{-7}mm.Hg.

Article by F. M. Penning and K. Nienhuis *Philips Tech. Rev.*, 11, Oct. 1949, 116-122

Automatic Regulation of Thermionic Emission

12/II

See Abstract No.: 17/IV

An Automatic Control of High Vacuum Systems

13/II

See Abstract No.: 39/II

An Improved Manostat and Manometer

14/II

See Abstract No.: 40/II

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Precautions to be Taken in the Use of the Philips Gauge — 15/II

France. This is a short report on experiments undertaken with a view of determining the influence of magnetic field strength on the sensitivity of the gauge. The permanent magnet usually provided for the gauge was replaced by an electro-magnet, which facilitated a change of field strength from 0-500 oersteds. The supply is a function of the field strength with V as the parameter. The pressure was held constant at 10^{-4}mm.Hg. It was found, that the sensitivity of the gauge was at its maximum for H = 370 oersteds, and V = 2,000. These are the exact data recommended by the inventor. If V is varied, the maximum of sensitivity will move along the hyperbolic curve in a graph with I as ordinate and H as abscissa. But it is difficult to produce fool-proof results in such experiments unless great care is taken that surface and metal composition of the electrodes are consistent.

Sommaire : Précautions à prendre dans l'usage d'une jauge de Philips.

Note by P. Leduc *Le Vide*, **4**, July-Sept. 1949, 684

22 — MEASURING PLANT (VACUUM) — 22

Mass Spectrometry — 16/II

United States. This is a survey article covering the progress of mass spectrometer design and techniques in the year 1949. Development work was concentrated on the analysis of liquids and their vapours rather than those concerning the analysis of gases. As a major development in the field of analytical applications the " International Standard "-method applied by Hindin, Grosse and Kirshenbaum is quoted. Design modifications to the inlet system aiming at overcoming errors caused by adsorption in the past resulted in placing the leak virtually within the ionization chamber and heating the system and the trap. In connection with applications in chemistry and physics, the work of Roberts and Nier is mentioned giving results of measuring packing fractions by means of the electrometric method. Finally, under the heading "Instrumentation ", Mariner's and Bleakney's design of a large mass spectrometer is referred to giving a resolution of 1 in 25,000 and employing crossed electric and magnetic fields. 79 references.

Sommaire : Une revue des développements concernant la construction et les applications du spectromètre de masse pendant l'année 1949.

Article by M. Shepherd and J. A. Hipple *Analyt. Chem.*, **22**, Jan. 1950, 23-5

Electron Microscope — 17/II

United States. Developments in 1949 are surveyed and emphasis is given to the increasing importance of the actual progress made in replica techniques. Some 350 electron microscopes are now in use all over the world, the majority of which are located in the U.S.A. One firm has started marketing a microscope-diffraction camera combination. Finally, electron diffraction is discussed but it is stated that very little is published on this subject and, in the view of the author, diffraction techniques have not been given the attention they deserve. 86 references.

Sommaire : Ou décrit les développements du microscope electronique pendant l'année 1949.

Article by F. A. Hamm *Analyt. Chem.*, **22**, Jan. 1950, 26-3

Electron Diffraction — 18/II

France. The basic laws underlying the functions ot electron diffraction apparatus are given and details of various camera designs are described. Examination of crystal surfaces by the transmission method and reflection method are discussed and comments added on the importance of electron diffraction examination techniques as compared with X-ray diffraction techniques. 10 references.

Sommaire : Les lois régissants les fonctions des appareils pour la diffraction des électrons.

Article by J. Ternision *Microtecnic*, **4**, Jan.-Feb. 50, 18-32

A Three-Stage Electron Microscope with Stereographic Dark Field, and Electron Diffraction Capabilities — 19/II

United Kingdom. An Electron Microscope is described in which a range of magnification between 1,000—100,000 times can be covered continuously by a means of an intermediate projector lens. Details of the design are given, in particular the construction of the microscope and the power unit used. The instrument can be used to obtain electron diffraction patterns. Photographs of micrographs are added.

Sommaire : On décrit un microscope électronique à trois étages, qui permet aussi des études au moyen de la diffraction des électrons.

Article by M. E. Haine, R. S. Page and R. G. Garfitt *J. Appl. Phys.*, **21**, Feb. 1950, 173-8

The Use of Spraying Methods and of Volatile Suspending Media in the Preparation of Specimens for Electron Microscopy — 20/II

United States. Two wholly volatile diluents, containing electrolytes and adjusted to normal pH, have been developed for use in forming spray drops. The use of volatile electrolytes makes it necessary to wash the specimen after the droplet patterns have been formed on the specimen screens. The following are some of the advantages claimed for the spraying technique : (I) Brief drying time of the droplets, (II) the possibility of comparing a particular composition of two closely similar suspensions, (III) the comparative ease in detecting differences between the structure of the specimens material and the substrate.

Sommaire : On discute les méthodes de vaporisation au pistolet dans la préparation des specimens pour le microscope electronique.

Article by R. C. Backus and R. C. Williams *J. Appl. Phys.* **21**, Jan. 1950, 11-15

Vol. 1 1951

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Preparation of Specimens for the Electron Microscope — 21/II
See Abstract No.: 8/III

A Low-Temperature Replica Method for Electron Microscopy — 22/II
See Abstract No.: 9/III

Uranium in Microscopy — 23/II
See Abstract No.: 12/III

Gilding the Lily — 24/II
See Abstract No.: 10/III

Seeing Molecules — 25/II
See Abstract No.: 11/III

The Electronic Telescope — 26/II

France. The general design principles of an electronic telescope are discussed. The objective is to improve the effectiveness of the conventional type used in astronomical work without having to increase the dimensions of the latter. The idea of the electronic version is based on apparatus developed by the Germans for application in aerial night fighting. Then, infra-red radiation produced an invisible image of the object on a screen from which electrons were emitted and accelerated *in vacuo*, eventually hitting a second fluorescent screen and thus forming a visible picture of the object. The design of the electron telescope provides for the initial image to be formed by the same means as in the case of an ordinary telescope. But instead of a photographic plate taking the picture, a vacuum tube is inserted carrying a layer of ceasium. Light falling on the ceasium layer causes the emission of electrons inside the tube. Their energy is increased in an electric field and their path controlled by a magnetic lens. Finally they impinge on a photographic plate inserted into the vacuum tube. 100 photons are required to eject 1 photo-electron and 1 photo-electron reduces 1 silver grain on the photographic plate. In the ordinary telescope 10,000 photons are required to produce the same effect, *i.e.*, the effectiveness of the electronic telescope is 100 times greater. The design of an ordinary telescope of equivalent performance would call for dimensions impracticable to build.
No details are given of the vacuum equipment required.

Sommaire: Le principe d'un modèle d'un telescope electronique est décrit.

Article by Anon. *Discovery* **11**, June, 1950, 172-173

A Large Dempster Type Double-Focussing Mass Spectrograph — 27/II

United States. Instruments of this type are well suited for use with hetero-energetic ion sources. Particularly the hot-spark type possesses great advantages in analytical work. The instrument described was built for the Wesleyan University. The dispersion of the instrument is 2.2 mm. or 1% mass difference. The theoretical resolution for a 0.001 inch principle slit is 7,000. The slit system provided for in this case effectively isolates the source region from the analysing region. Each region is pumped separately. Details of the pumping system are given. A diagrammatic sketch is shown of the four-chamber design which permits insertion and removal of photographic plates without breaking the vacuum.

Sommaire: On donne des détails sur un spectromètre de mass à double mise au point.

Article by H. E. Duckworth *Rev. Sci. Instrum.* **21**, Jan. 1950, 54-59

A Recording Mass Spectrometer — 28/II

United States. A detailed description is given of a recording mass spectrometer setting out the principles of its operation and the potential fields of its application. The normal sensitivity of the instrument is 1 part in 10,000. If necessary this can be adjusted to 1 part in 100,000. The scanning range covers masses of 1 to 300. If required, this range can be extended to cover masses up to 400. Main applications: The analysis of gases, liquids and–in certain cases–solids.

Sommaire: L'article contient des détails sur un spectromètre de masse en registreur.

Article by R. M. Ilfeld *Instrumentation*, **4**, Spring 1950, 20-22

Mass Spectrometer for Leak Detection — 29/II
See Abstract No. 41/II

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

Particle Size of Evaporated Gold — 30/II

See Abstract No.: 15/III

Measurement of Appearance Potentials of Ions Produced by Electron Impact, Using a Mass Spectrometer — 31/II

See Abstract No.: 6/IV

The Apparent Size of Object as Observed in the Electron Microscope — 32/II

United States. Certain experiences in connection with work under the electron microscope seemed to indicate that there is a difference in the image size of small objects, depending on whether they are shadowed or unshadowed. A systematic investigation into the phenomenon using Dow Latex particles, known for their regularity of size, had the following results: The shadowed balls (shadowing at an angle of 10°) measured 2840Å ± 50Å, whereas unshadowed balls measured 2590Å ± 50Å. The most probable explanation for this effect is that a charge differential exists between the polystyrene sphere and its surroundings. If the spherical particle is negative, the potential gradient around it would constitute a negative electron lens and act on electrons passing through or near the particle. This would result in the size of the image of the sphere being smaller than would be expected, if the sphere were not charged. Shadowing, however, with a conducting film (platinum-palladium in this instance) will reduce any charge differential between the particle and its surroundings and consequently eliminate the lens effect. Details of the experimental procedure and possible other explanations of the phenomenon are given.

Sommaire: C'est une étude sur la grandeur apparente d'un object examiné au microscope electronique.

Article by S. F. Kern and R. A. Kern *J. Appl. Phys.* **21**, July, 1950, 705-707

26 — MATERIALS — 26

Porcelain, Its Manufacture and Uses — 33/II

United Kingdom. The industrial importance of porcelain is discussed, with particular reference to its uses in the field of electrical engineering. Present-day scientific research is essentially directed at producing materials of greater toughness and lower thermal expansion. But it is not only the composition of the paste that counts. Its physical condition is no less important. A machine has recently been developed which finally shreds the plastic body under vacuum and then re-consolidates it producing a de-aerated material of much improved stability and consistency of quality.

Sommaire: La qualité des porcelainés industrielles est améliorée en employant un appareil faisant usage de l'action du vide

Article by Anon. *Times Indust. Suppl.*, July 1950, 14

Behaviour of Refractory Oxides of Metals, Alone and in Combination, in Vacuo at High Temperatures — 34/II

United States. The behaviour of carbon, beryllia, magnesia, zirconia, thoria, molybdenum, tungsten and certain combinations of the same have been investigated in a vacuum induction furnace for the effects of temperature and time on linear shrinkages, densification and loss in weight of dry-pressed compacts. In addition, the effect of time and temperature *in vacuo* on surface-to-surface stability was studied. The results are given in tabulated form. It has been found that the limiting factor for many refractories at high temperature *in vacuo* is not their melting point but their stability when in contact with other substances.

Sommaire: Une étude sur le comportement des oxydes réfractaires des métaux dans le vide.

Article by P. D. Johnson *J. Amer. Ceramic Soc.* **33**, 1.5.1950, 168-171

28 — APPARATUS COMPONENTS — 28

The Design of a Large Vacuum Valve for Application to an Electric Furnace — 35/II

United Kingdom. A process has been developed by the National Physical Laboratories for the production of high purity iron, the last step of which requires the de-oxidation of the iron in a vacuum high-frequency furnace. For the purpose of facilitating the manipulation of hydrogen and the detection of leaks in the pipe line connecting the furnace with the pumps a valve had to be inserted in the line (8 inches diameter). The head of the valve has a travel of 12 inches. The sealing of the valve head is effected by pressure rings, and the sealing of the valve spindle and the housing by a Wilson seal. The valve is mounted in a right angle elbow of the pipe line and, therefore, has little adverse effect on the pumping speed.

Sommaire: Description d'un robinet à vide à grand débit pour un four électrique.

Article by W. F. Arkins and G. C. H. Jenkins, *Machinery*, 9.3.1950, 351-52

A Low-Impedence High Vacuum Valve — 36/II

Australia. Description and sketch of a " Straight-Through "-type valve are given as an example of modern design. The sealing plate is operated by a crankshaft and rises vertically above the seat without rotating

Vol. 1 1951

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

up to a height slightly greater than its radius. If the sealing plate is lifted up beyond that point, it swings into the vertical plane. This arrangement is satisfactory as long as the valve is in a vertical position. In any other position, however, the sealing plate may jam and for this reason a kick plate is fastened to the base which will ensure smooth working of the valve in any position whatsoever.

Sommaire : Un dessin d'un robinet à vide qui ne veut pas empêcher le passage de gaz.

Note by R. I. Garrod *J. Sci. Instrum.* **27**, July 1950, 205

A Motor for Use in Vacuum Systems

37/II

United States. It is often desirable to arrange for a motor to operate inside a vacuum chamber. One of the greatest difficulties in that respect is the outgassing from the field coils, the rotor and the bearings, even at temperatures below 100°C. A design is described which overcomes these difficulties. It makes use of a solid armature motor whose field coils are hermetically sealed in a brass can. The leads to the coils are connected through Korvarglass sealed lead-ins. The armature is held together with soft metal which can easily be turned to a diameter giving sufficient clearance between the armature and the cased-in field. After completion the armature is subjected to a special dip treatment and the ball bearings washed with acetone. Octoil is used as a lubricant. A motor of this design was used in an evaporation plant and mounted inside the bell jar. The maximum temperature after two hours running did not exceed 35°C.

Sommaire : On décrit un moteur électrique construit pour être employé dans une cloche à vide.

Note by C. P. Butler and F. E. Carpenter *Rev. Sci. Instrum.* **21**, Jan. 1950 103

Cutting Flat Vacuum Gaskets on a Lathe

38/II

United States. A simple method is described for the cutting of flat vacuum gaskets (of rubber, neoprene, silastic, etc.) on a 10 inch lathe without gluing the gasket sheeting onto a face plate.

Sommaire : Conseils sur la façon de couper des garnitures au tour.

Note by R. B. Heller *Nucleonics*, **6**, Feb. 1950, 81-82

29 — MISCELLANEOUS APPARATUS — 29

An Automatic Control of High Vacuum Systems

39/II

United States. An ionization gauge and indicator unit is described incorporating a fully automatic circuit for the measurement and control of high vacuum systems. After an initial adjustment no further attention from the operator is required. The pressure is indicated on a large meter calibrated directly in mm.Hg and the required multiplication of readings by a system of panel lights. An automatic scale-changing mechanism is provided to adjust the sensitivity of the indicator keeping the meter readings between 6 and 100% of full scale at all times. The same mechanism is suitable for interlock functions involving the control of other apparatus at predetermined pressure levels. Should the pressure in the system rise to excessive values, the circuit switches off automatically.

Sommaire : Une jauge à ionisation incorporant un circuit permettant la mesure et le contrôle des systèmes a vide.

Article by J. W. Clark and G. H. Witts *Electronics* **23**, June 1950, 108-110

An Improved Manostat and Manometer

40/II

United States. A manostat is described for use in connection with vacuum distillation which, it is claimed, is better adapted to precision vacuum fractionation than designs in use hitherto. Its essential feature is a magnetic operated screw for adjustment to any pressure within a wide range. The screw made of stainless steel is placed inside the manometer glass tube and terminates at the top in a soft wire vane. This vane can be turned (for adjustment) by rotating the magnet placed outside the glass envelope. A stainless steel nut travelling on the screw carries a tungsten wire which makes contact with the mercury at the desired pressure, closing an electrical circuit *via* a platinum wire permanently in contact with the mercury in the tube. An electronic relay in this circuit operates a solenoid which governs the position of a plunger opening or closing an orifice in the system as the case may be. The lowest pressure at which the manostat was operated was 3 mm.Hg, the highest pressure 523 mm.Hg.

Sommaire : On donne des détails sur les améliorations d'un " manostat "

Note by W. P. Ratchford and M. L Fein, *Analyt. Chem.*, **22**, June 1950, 838-9

Mass Spectrometer for Leak Detection

41/II

United States. A portable leak detector of the mass spectrometer type, as used in connection with the gaseous diffusion plant at Oakridge for the concentration of uranium isotopes, is described and pictured diagrammatically. It is imperative that no appreciable amount of air with normal water content penetrates into the plant as otherwise it would facilitate the formation of H_2F_2. Therefore, the maximum in-leakage specified as permissible determining the sensitivity required from the instrument is 1/100th of a standard cubic foot per hour per 1,000 cubic feet of process equipment volume. This is equivalent to a pressure build-up of 8 microns per hour at a high vacuum. Helium is used as a tracer gas. The mass spectrometer described has been developed by Professor Nier in 1942.

Sommaire : Description du spectromètre de masse utilisé à Oakridge pour la detéction des fuites.

Paper by Z. G. Deutsch and F. Raible, *Chem. Engng.*, **57**, April 1950, 279-284

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

An Audible Vacuum Leak Detector

42/II

United Kingdom. Description and sketch are given of a leak detector producing aural signals. The essential element of the device is a two-electrode discharge tube inserted in the vacuum line. The characteristics of the discharge change with the pressure and nature of the gas. If the discharge tube is made the variable factor in a relaxation oscillator circuit, the oscillation frequency made audible over the loud speaker will vary in accordance with the pressure changes occurring in the vacuum line. The instrument covers the pressure range of 10^{-1} mm.Hg.

Sommaire : Un systeme sonore pour la détection des fuites.

Note by J. T. Lloyd *J. Sci. Instrum.* **27**, March 1950, 76

Method of Handling and Storing Liquefied Gases

43/II

See Abstract No. : 7/IV

Vol. 1 1951

III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

Metallising by High Vacuum 1/III

United States. The comparative merits of three methods of metallising plastics are discussed : (1) Metallising by vacuum evaporation. (2) Silver reduction. (3) Electro-plating. Production equipment and lay-out are discussed and tables are added illustrating the findings of the author. Two of these tables are reproduced here :—

Comparison of Metallising Methods

Technique	*Vacuum Metallisation*	*Silver Reduction*	*Electroplating*
Appearance	Excellent	Excellent	Very Good
Utility (*)	Very Good	Very Good	Excellent
Unit Cost	$.03	$.07	$.14
Remarks	Physical details maintained. Service utility of finish dependent upon protective lacquers used. Brightness of finish unaffected by ageing.	Physical details maintained. Service utility of finish dependent upon protective lacquers used. Brightness of finish affected by ageing.	Physical details blurred. Service utility of finish excellent. Brightness of finish may be affected by ageing.

(*) Evaluation of the factors that determine the functional value of the item, including permanence of finish under service conditions, weight, protection afforded to the plastic item, etc.

Coating Cost Comparison

	Silver Reduction	*Vac. Metallization*
Production volume per day	300 gross pieces	300 gross pieces
Initial capital equipment investment	$20,000	$28,000
Labour required	28 man days	9 man days
Total direct costs	$0.0013 per unit	$0.0003 per unit

Article by M. Schneider *Modern Plastics* April, 1950 **135**, 138-140

Sommaire : On compare le procédé de métallisation des matières plastiques par évaporation sous vide avec le procédé de réduction chimique des sels d'argent et du plaquage galvano-plastique.

Symposium on Thin Films 2/III

United States. The Armour Research Foundation (Illinois Institute of Technology) organised a symposium on thin films, which took place between the 8-10th June 1950. All aspects of thin film production, structure and behaviour were discussed. Evaporation procedures and the behaviour of various metals like germanium, aluminium and silver in such processes were described. Reports were given on oxide film formation, pin holes and adhesion. Thin films of organic materials were illustrated by the " dark " and " bright " series of interference colours produced by a wedge film of barium stearate on glass. Finally the latest developments regarding electron microscopes were discussed.

Article by F. A. Hamm *Analyt. Chem.*, **22**, July 1950, 958-9

Sommaire : Compte-rendus d'un colloque sur les couches minces tenu aux Etats-unis du 8 au 10 Juin 1950.

Improvements in and Relating to Non-Reflecting Optical Bodies 3/III

United Kingdom. The invention concerns procedures of coating optical bodies such as lenses and prisms designed for a high light transmission, and aiming at reducing the internally reflected stray light to a minimum. It is proposed to deposit on the surface of the body in contact with air films of different thicknesses with the object of suppressing reflection of light of unlike wavelengths.

British-Thomson Houston Co. Ltd., *Brit. Pat.* 634,776

Sommaire : Un brevêt sur un procédé de déposition des couches anti-réfléchissantes sur des pièces d'optique.

Method of Eliminating Moisture from the Surfaces of Moisture Absorbent Material on Which Metal is to be Deposited by Thermal Evaporation in Vacuo 4/III

See Abstract No. 24/III

On the Growth of Metallic Crystals from the Vapour Phase 5/III

United States. There are four known ways of growing crystals : (1) From the melt; (2) from solution; (3) metastable crystals in contact with growing crystals in a continuous solid and (4) from the vapour. There is comparatively little known about method (4) but its possibilities have lately attracted much interest. Details are given of experiments concerned with the growth of metallic cadmium and zinc crystals. A vacuum chamber was used with a crucible made of molybdenum. The temperature of the evaporating source and

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

collector plates has been varied and data recorded for different pressures of the nitrogen gas atmosphere. The optimum conditions for the growth of large single crystals of both these metals are tabulated as follows :—

	Temperature of Vapour Gun Source	*Partial Pressure of Vapour Source*	*Temperature of Collecting Surface*	*Pressure of Nitrogen Atm.*
Cadmium	310-321°C	1×10^{-1}mm.Hg.	305 ± 3°C	1×10^{-2}-5 $\times 10^{-2}$mm.Hg
Zinc	409-419°C	2×10^{-1}mm.Hg.	410 ± 3°C	1×10^{-2}-3 $\times 10^{-2}$mm.Hg

Sommaire : Sur la "croissance" de cristaux métalliques par la condensation de leurs vapeurs.

Article by G. R. Keeping Jr. *J. Appl. Phys.*, **21**, March 1950, 260-261

The Thermal Etching of Single Crystals of Cadmium

6/III

See Abstract No. : 12/I

Manufacturing Metallised Picture Tubes

7/III

United States. The production of aluminium-backed screens introduces two more steps into the conventional production procedure of picture tubes, *i.e.*, the application of a film carrier and the deposition of the actual metal film on the same. When the processing of the phosphorus screen is finished the film carrier is applied by placing a shallow level of water over the screen. A few drops of a specific organic solution are added. This material hardens in the water and forms a membrane on the water surface. The water is then removed by mechanical means and the membrane comes to rest on the phosphorus screen ready to receive the metal coating. Aluminium is the metal ordinarily chosen for this process but magnesium, silver or chromium could also be used. Details are given of the effect of varying thicknesses of this backing layer on the brightness performance of the phosphorus screen. The figure of 6.8×10^{-6} in. is given as the preferred thickness. The evaporation plant required for aluminizing tubes on a mass production basis is described in detail.

Sommaire : Détails sur la fabrication d'écrans recouverts d'aluminium pour des tubes de télévision.

Article by E. R. Ewald *Electronics*, **23**, Feb. 1950, 76-7

Vol. 1 1951

Preparation of Specimens for the Electron Microscope

8/III

United Kingdom. A review of the March issue of the "Journal of the Royal Microscopical Society" which is devoted entirely to an account of the techniques of specimen preparation for use in the electron microscope. According to the reviewer the publication is a comprehensive and representative compilation of all preparative methods and related techniques. It does not deal with the operation and maintenance of the electron microscope itself nor does it deal with the outcome of research facilitated by this instrument.

The title of the compilation is "The Practice of Electron Microscopy", the author is D. G. Drummond.

Sommaire : Revue d'une édition spéciale du journal de la "Royal Microscopical Society" entièrement consacrée à un exposé des techniques de préparation des spécimens.

Review by V. E. Coslett *Nature*, **165**, 24. 6. 1950, 1009

A Low-Temperature Replica Method for Electron Microscopy

9/III

United States. This is a description of a replica method suitable for electron microscopy of aqueous substances in the frozen state. It consists of placing the specimen on glass in a vacuum chamber in contact with a copper block which is pre-cooled to liquid air temperature. After evacuation the temperature is raised temporarily to–60° to–90°C by means of radiant heat to allow ice crystals which may have condensed on the specimen from the atmosphere to sublime and at the same time to secure a surface etch. Thereafter the specimen is shadowed with Cr and a supporting film of Si evaporated from a BeO crucible is deposited on the surface. Finally the cold block is removed from the vacuum chamber the glass is detached and the surface film is floated on water after the specimen has warmed up to room temperature.

Sommaire : Application de la techniques des répliques en microscopie electronique aux spécimens aqueux congelés.

Note by C. E. Hall *J. Appl. Phys.*, **21**, Jan. 1950, 61-62

Gilding the Lily

10/III

United States. The application of metallic shadow casting techniques to biological specimens is discussed. The purpose of the technique is to render biological objects or replicas opaque to facilitate closer study. The technique is similar to the one used in the metallurgical field, the main difficulty being to cater for large masses of tissue and entire embryos. Details are given of the plant and of the materials used for evaporation. It is stated that the technique shows up the surface texture of embryos clearly and that the study of dissections of normal or pathological embryos will greatly benefit from it.

Sommaire : Une courte note sur l'application de la technique des ombres aux spécimens biologiques.

Note by Anon. *Industr. Engng. Chem.*, **42**, March 1950, 16-18A

Seeing Molecules

11/III

United States. The technique of resinography is discussed, a branch of plastics chemistry akin to metallography and crystallography. If a plastics specimen is broken up, the chances are that the cleavage occurs between molecules rather than between atoms within a molecule.

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Examination of the fracture at sufficiently high a magnification should then show details of the structure of the molecules constituting the plastic. In practice, the specimen is prepared by chilling the plastic until it is brittle, breaking it up and casting a negative replica on a gelatine or polyvinyl alcohol film. Then, silica is evaporated *in vacuo* onto the replica and the film is dissolved away leaving a positive replica ready for use in the electron microscope. Results of the examination of silicone rubber specimens prepared in this manner are given.

Sommaire : On discute la préparation des spécimens en matière plastique pour l'étude au microscope électronique.

Article Anon. *Chem. Industr.*, **66**, May 1950, 664-665

Uranium in Microscopy

12/III

United States. Uranium is recommended as a material for shadow casting in connection with the preparation of specimens for examination in the electron microscope. It is suggested to make use of this metal on account of its high atomic number and great density, where specimens of light weight elements are to be investigated as these are often only showing up dimly on the electron microscope plate. Uranium is claimed to be superior to chromium in this application.

Sommaire : On recommande l'usage de l'uranium pour la technique des ombres.

Note by Anon. *Chem. Industr.*, **66**, March 1950, 406

The Structure of Obliquely Evaporated Films and Their Influence on the Formation of Sub-Microscopic Surface Irregularities

13/III

Germany. The factors determining the surface structure of ' shadow-cast ' films are discussed. The condition of the surface is not governed by the material of the substrate but by the structure of the evaporated film. Thickness of the deposit and angle of incidence of the molecular rays is important. The latter should not be less than 20°, otherwise a misleading ' apparent ' roughness of the film surface may result. In addition, long ' shadows ' may cover up details of the specimen under observation in the electron microscope which are valuable to the investigation in progress. Granulation of gold films occurring in high-density electron beams can be stopped by applying a thin film of varnish after deposition.

Sommaire : Une étude sur les facteurs affectants la structure des couches employées dans la technique des ombres.

Article by H. Koenig & G. Helwig *Optik*, **6**, Feb. 1950, 111-124

Surface Tension Effects on Oriented Thin Silver Films

14/III

United States. The structural changes of oriented thin silver films have been investigated which had been deposited on rock salt at 200°C by vacuum evaporation and subsequently heat-treated at high temperatures. Films of four different thicknesses were prepared: (1) From 100 to 500 Å; (2) from 500 to 1000 Å; (3) from 1000 to 1500 Å, (4) from 1500 to 2000 Å. At a thickness up to 1000 Å a marked agglomeration was noticed in the film. At thicknesses between 1000 and 2000 Å rectangular holes appeared in the film, the edges of which lined up with the 110 directions of the rock salt. Very thin films will break up into small agglomerated regions, if the temperature is raised to sufficiently high a value. These effects are attributed to the effects of surface tension forces due to the re-adjustment of the oriented surface.

Sommaire : Changements de structure des couches minces d'argent sous l'influence de la chaleur.

Article by G. E. Johnson *J. Appl. Phys.*, **21**, May 1950 449-453

Particle Size of Evaporated Gold

15/III

United States. There is a wide-spread belief that gold deposited by evaporation constitutes a smooth continuous film which granulates as a result of exposure to an intense electron beam as shown in shadowed electron micrographs. This is not borne out by the facts established in an investigation carried out and reported here. It was found that gold deposits contained about 5% tungsten. Films of this type showed the characteristic matter appearance. However, when the percentage of tungstic oxide was reduced by flushing the system during evaporation by purified nitrogen, for instance, the gold appeared as discreet particles. This proves that granulation of gold films is subject to the composition of the evaporated material and not produced when exposed to electron bombardment.

Sommaire : On examine la granulation des couches d'or.

Article by P. G. Wilkinson & L. S. Birks *J. Appl. Phys.*, **21**, Jan. 1950, 60

The Photo-Conductivity of Bismuth Sulphide and Bismuth Telluride

16/III

United Kingdom. In a U.S. patent application it has been suggested that bismuth-sulphide and bismuth-telluride may be photo-sensitive up to a wave length as long as 7 microns. The authors have come to a different result in the course of experiments with photo-conducting and photo-voltaic layers produced by vacuum evaporation from Bi_2S_3 and Bi_2Te_3. Two graphs are shown one giving the photo-conductivity spectrum of bismuth-sulphide and the other of bismuth-telluride. Both show a sensitivity considerably less than that claimed in the American publication. In the case of Bi_2Te_3 response did not exceed about 4μ. Oxidation of this material can produce an extension of the spectral sensitivity by 1μ.

Sommaire : ' Photo-conductivité ' des couches de sulphure et de ' tellurure,' de bismuth.

Letter by A. F. Gibson & T. S. Moss *Proc. Phys. Soc.*, **63, A**, 1.2.50 176-177

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Thermionic Emission of Thin Films of Alkaline Earth Deposited by Evaporation 17/III
See Abstract No. : 15/IV

Direct Optical Measurements of the Length of Organic Molecules 18/III
See Abstract No. 14/I

Experimental High Megohm Resistors 19/III
See Abstract No. : 8/I

Evaporation and Outgassing in an Inert Atmosphere 20/III
See Abstract No. : 11/I

The Apparent Size of Objects as Observed in the Electron Microscope 21/III
See Abstract No. : 32/II

31 — CATHODIC SPUTTERING — 31

Symposium on Thin Films 22/III
See Abstract No. : 2/III

Cathodic Vacuum Etching of Metals 23/III
Article by D. M. McCutcheon & W. Pahl
Metal Progress Nov. 1949 674-679
see also Anon. *Machinery*. 11.5.1950, 668

United States. In 1948 a method of cathodic vacuum etching was made known which will yield information on the structure of material in cases where conventional methods of etching produce little result. This article gives a summary of the principles of this method, the scope and the results obtained in some of its applications. Illustrations are given among others of the structure of an austenitic chromium-nickel steel and an alloy of the cobalt-nickel tungsten-iron type.

Sommaire : On donne des détails sur le ' décapage ' par la désintégration cathodique.

36 — DRYING — 36

Improvements Concerning Methods of Eliminating Moisture From the Surfaces of Moisture Absorbent Sheet Material on which Metal is to be Deposited by Thermal Evaporation in a Vacuum 24/III

United Kingdom. An arrangement is described which consists of an inner and an outer chamber with slots in the communicating walls through which sheet material is being conveyed, to pass under a radiation heat source positioned in the inner chamber. Both chambers are evacuated jointly by a vacuum pump connected to the inner chamber. Thus, pressure in the inner chamber is smaller than in the outer chamber, and vapours which come off the sheet material whilst subjected to radiation, will not penetrate into the outer chamber, where the material is cooled down before it reaches the take-up role.

Sommaire : Description d'un assemblage à deux compartiments pour la dessication préliminaire sous vide des matières plastiques en forme de feuilles.

P. Alexander
Brit. Pat. 639,099

Antibiotics Developments 25/III

United States. A detailed account of procedures adopted for the fermentation and finished drying of penicillin and streptomycin. Both these processes call for the application of vacuum but each antibiotic has to be considered individually, when it comes to laying out the production procedure in detail. Details are given of a vacuum drum-drying (pilot) plant with special features to suit the purpose. Feeding to and collecting from the apparatus can be effected without disturbing the vacuum and thus affecting the quality of the product. Special means are provided to ensure proper control of the pressure required for the drying operation. Finally, the machine is pre-sterilised before each drying operation by being ' washed ' with specially filtered air.

Sommaire : On discute la fermentation et la purification (ainsi que le séchage final) de plusieurs antibiotics. Des détails sur le développement d'un appareil pour le séchage final sous vide sont aussi donnés.

Article by W. B. Fortune, S. L. McCormick, H. W. Rode-Hamel & J. J. Stefaniak.
Industr. Engng. Chem., **42**, Jan. 1950, 191-198

Vol. 1 1951

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

37 — METALLURGICAL PROCESSES — 37

Modern Practice in Investment Casting — 26/III

United States. All aspects of this production technique are discussed : preparing the investment and the mould, gating practice and, melting and pouring. In preparing the investment, it is stressed, particle size and moisture must be closely controlled. Details of the vacuum treatment are given. The optimum vacuum to ensure complete removal of all bubbles after mixing has been found to be approximately 29 inches. Finally four methods of pouring are mentioned : (1) Static; (2) centrifugal; (3) under high pressure; (4) *in vacuo*, but no details are given of method (4).

Sommaire : On décrit les différents aspects de la méthode de moulage à la cire perdue.

Article by J. Brown *Foundry*, Dec. 1949, 93

Improvements in Processes and Apparatus for the Treatment of Hafnium, Zirconium, Titanium, Thorium and their Alloys, in the Molten State — 27/III

United States. This patent covers the production of shaped metallic articles consisting of hafnium, zirconium, titanium or thorium and their alloys. In particular, it refers to the processing of such metal in the molten state *in vacuo* or in a noble gas atmosphere. It is proposed to carry out such treatment in apparatus, where the surfaces in contact with the molten material consist of a material belonging to the group of carbons, graphites or high melting carbides. Thus, it is claimed, subsequent embrittlement can be stopped by precluding impurities from entering the processed article.

Sommaire : Une façon d'empêcher que les métaux rares soient cassants.

W. J. Kroll *Brit. Pat.* 633, 117

Design of a large Vacuum Valve for Application to an Electric Furnace — 28/III

See Abstract No. : 35/II

Determination of Carbon in Ferrous Alloys — 29/III

United States. Various methods employed in different laboratories for the determination of carbon in ferrous alloys are described. Among others details are given of the so-called low-pressure combustion method. It consists essentially of burning the sample in an oxygen atmosphere at 160 mm.Hg after degassing the crucible, freezing the carbon dioxide in a cold trap, pumping off the excess oxygen and vapourising the dioxide in a known volume. A precision of 0.0002% or better can be obtained from a 0.5 gram sample. The method is slow but is claimed to be the most precise for determining carbon content of low-carbon alloys.

Sommaire : Description de la méthode de combustion à pression réduite pour le dosage du carbone dans les alliages (de fer)

Article by W. G. Guldner *Analyt. Chem.*, **22**, March 1950, 486

Analysis of Oxygen in Titanium — 30/III

United States. A vacuum fusion method is described which represents a modification of the conventional procedure but makes use of the standard vacuum fusion apparatus. The actual analysis is carried out in accordance with known low-pressure fractional freezing methods. The method is used in connection with the analysis of titanium–oxygen alloys containing up to 0.5% oxygen.

Sommaire : On décrit une modification de la méthode de fusion sous vide pour le dosage de l'oxygène dans le titane.

Article by G. Derge *J. Metals*, Oct. 1949 31-33

A Vacuum Fusion Furnace for the Analysis of Gases in Metals — 31/III

United States. The furnace is made of glass and quartz, the crucible is made of graphite. The envelope of the furnace is sealed to a two-stage mercury diffusion pump by means of a 1 in. diameter glass tube. The graphite crucible is located in a quartz tube of 2 in. diameter and is embedded in graphite powder. The furnace is cooled with a high velocity air stream which is directed uniformly about the walls of the furnace by means of a glass funnel of special design. The furnace described has been in constant use for 2 years and in some instances, a heat of 2,400°C has been applied to the processed material for an uninterrupted period of 6 hours at a time.

Sommaire : Un four en verre pour l'analyse des gaz dans les métaux par la fusion sous vide.

Article by W. G. Guldner & A. L. Beach *Analyt. Chem.*, **22**, Feb. 1950 366-367

A Vacuum Furnace for Use in the Temperature Range of 1000°-2000°C — 32/III

United States. A vacuum furnace is described, designed for investigation work of a physico-chemical nature in the temperature range of 1000°-2000°C. The crucible is made of molybdenum and the heater from tungsten wire in the form of a cylinder. Radiation shields are provided, made from molybdenum sheet of 0.2 mm. thickness. All three components are suspended in a manner which counteracts sagging at high temperatures.

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

The envelope of the furnace is made of pyrex glass of 70 mm. diameter, with an observation window at the end of the tube. The furnace is run at a pressure of approximately 10^{-3} to 10^{-4}mm.Hg and the pumping system should provide for a speed of 10-20 litres/second at 10^{-4}mm.Hg. The heater consumes 35 amps at 40 volts to maintain a temperature of 2000°C. It takes about 15 minutes to reach this temperature.

Sommaire : Description d'un four à vide pour les températures élevées.

Article by J. W. Tomlinson & J. O. M. Bockris *Rev. Sci. Instrum.* **21**, June 1950, 507-508

Apparatus for the Preparation of Metals with an Exactly Known Content of Impurities

33/III

Holland. A melting apparatus is described which facilitates the study of the effect of carbon, oxygen and nitrogen separately on iron and steel. Melting is effected by induction heating. The source of supply is a 14 kw. generator at a frequency of 330,000 c/s. . The melting chamber is made of glass (water-cooled), the crucible of pure aluminium oxide. In preparing the metal a vacuum of the order of 10^{-5}mm.Hg is applied. 99.9% pure carbonyl iron prepared in this apparatus contains no more than 0.001% of carbon + oxygen + nitrogen. The apparatus provides for carbon to be added in the form of pure graphite and for oxygen and nitrogen to be added in the gaseous state. Details of the apparatus, the method of preparation and admixing the various constituents are given.

Sommaire : Description des appareils nécessaires pour l'étude des effets du carbone, de l'oxygène et de l'azote sur le fer et les ăciers.

Article by J. D, Fast *Philips Techn. Rev.*, **11**, Feb.1950, 241-244

Low-Pressure Distillation of Zinc, from Al-Zn Alloys

34/III

United States. A report is given of a very thorough investigation into the processing details and potential advantages of low pressure separation of zinc from aluminium. The vacuum distillation unit used for the purpose consists essentially of an induction furnace, a condenser, the vacuum system and a power convertion unit. Power is supplied from a 50 kw.3000 c/s motor-driven alternator. The unit was operated at a pressure of the order of 0.06 mm.Hg. Pressure measurements were taken with a McLeod Gauge in the actual vacuum system and in the furnace with a thermocouple gauge. To arrive at a proper assessment of the efficiency of the equipment pure zinc was distilled in the first instance at different temperature levels and it was found that the evaporation rate was more directly related to the power input than to the metal temperature. The actual separation tests were carried out for two different types of material, (*a*) alloys prepared from aluminium and (*b*) from aluminium scrap. The physics of the process are discussed at length.

Sommaire : Les principes et la technique de la distillation sous vide du zinc des alliages aluminium-zinc.

Article by M. J. Spendlove & H. W. St. Clair *J. Metals*, Sept. 1949, 553-560

Production of Malleable Zirconium

35/III

United States. A description is given of a pilot plant capable of producing 300 lbs. of zirconium sponge a week. The production process consists essentially of 6 steps : (1) Arc-furnace smelting of the zirconium ore with the addition of carbon to produce an impure carbide containing approximately 80% zirconium. (2) Chlorination of the carbide to produce raw zirconium tetrachloride. (3) Purification and densification of the chloride in a hydrogen atmosphere. (4) Reduction of the purified dense chloride with fused magnesium in a helium atmosphere. (5) Separation of the excess magnesium and magnesium chloride from the zirconium sponge by vacuum distillation. (6) Melting the zirconium sponge in a split tube graphite resistor furnace under a vacuum. Details are given of the equipment required and the times taken by each individual process.

Sommaire : On décrit la préparation du zirconium sous forme d'éponge

Article by W. J. Kroll & W. W. Stephens *Industr. Engng. Chem.*, **42**, Feb. 1950, 395-398

38 — DISTILLATION — 38

High Vacuum Distillation

36/III

United States. A survey of developments covering the period following the high vacuum symposium held in Cambridge, Mass., in Autumn 1948 to the end of 1949. To adequately describe the trend of developments in the field of vacuum distillation the author suggests the use of a new term " Mega-Vacuum ", to indicate the modern requirements of transporting large masses of gases at a pressure level below that previously required in industry but higher than what is ordinarily termed high vacuum. Modern plant of this description calls for a vacuum " engine " as distinct from a vacuum pump.

Generally speaking, design is concentrated on the provision of still larger, quicker, higher and cheaper vacua. Commenting on detailed development the author mentions the falling-film still, the centrifugal still, the short-neck boiling-point still and fractionating columns with a rotary cooled stirrer within a heated tube. 59 references.

Sommaire : L'auteur commente sur les progrès effectués dans la technique de distillation sous vide pendant l'année 1949. Il attire l'attention sur la necessité d'avoir un vide meilleur, plus rapidement et à meilleur marché, et aussi d'un plus grand volume.

Article by K. C. D. Hickman *Industr. Engng. Chem.*, **42**, Jan. 1950, 36-38

Vol. 1 1951

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

Manufacture of Mononitrotoluene

37/III

United Kingdom. A booklet containing four lectures published under the auspices of the Institution of Chemical Engineers. Design problems and their solution are given emanating from the construction of chemical processing plant and much space is devoted to the tackling of vacuum problems, in particular vacuum distillation. The processes in question are carried out in a pressure range of 10-50 mm.Hg. Calculations are given of batch stills, continuous stills, pressure losses in vacuum lines and considerations on the assessment of steam ejector capacity required.

Sommaire : Conception d'un appareil de chimie employant la distillation sous vide.

Booklet by J. M. Coulson & F. E. Warner, *Inst. Chem. Engrs.*, Special Print, 55 pages

A Simplified Design for a Molecular Still of the Horizontal Type

38/III

United Kingdom. Modifications are reported of a horizontal type still originally designed by P. Meunier & Y. Raoul. A disadvantage of the original design was the necessity of tilting the apparatus during the operation to ensure proper flow of the liquid along the condenser. The new design provides for a ground joint between the vacuum line and still which allows for a standard tilt to be maintained during the whole of the operation. The condenser is positioned 1 cm. off centre from the axis of the envelope.

Sommaire : Un modèle simplifié d'un appareil à distillation moléculaire du type horizontal.

Letter by C. C. Smith & R. Matalon *Nature*, **165**, 15.4.1950, 613

Distillation Process for Fats

39/III

United Kingdom. A method is described of treating animal fats obtained as by-products from the slaughter of pigs and cattle. The process has been developed in U.S.A. and is based on techniques applied to the refining of petroleum. Essentially, the plant consists of a series of stainless steel fractionating distillation columns with bubble-cap trays. The plant is operated under a high vacuum. Initially the crude fats and oils are being cracked at high temperature. Subsequently, the crude stock flows through a series of towers and fractions of increasing boiling point are being removed. Each tower is maintained at a different level of vacuum and temperature and operates at a different reflux ratio. Fractionation into carbon groups of varying chain length is effected without separating compounds of the same carbon number. The operation of this plant is expected to have a marked influence on the production of wax mixtures, soaps, polishes and similar preparations in this country.

Sommaire : L'application industrielle de la distillation sous vide au traitement des graisses animales.

Article by Anon. *Times. Industr. Suppl.*, Feb. 1950, 31
See also : *Chem. & Drugg. Export Rev.*, **10**, April 1950, 54-55

Defatting Distillation

40/III

United States. Details are given of a procedure known as the Vio-Bin process for desiccating and defatting tissues. The process is a combination of azeotropic distillation and solvent extraction. In the past extraction of fat from fresh materials by means of a solvent has not been satisfactory because of the high water content, but, if a solvent is used which forms an azeotropic mixture with water in the tissue, the water can be removed by distillation under reduced pressure and this eliminates the danger of denaturation due to heat. Among others pancreas and liver are handled in the process yielding enzymatically active products.

Sommaire : Une note sur le procédé " vio-bin " de dessication et de dégraissage des tissus.

Note by R. L. K. *Industr. Engng. Chem.*, **42**, June 1950, 14A-18A

Multicolumn Reflux Still

41/III

United States. A short description is given of a multi-column counter-current molecular still designed by S. L. Madorsky of the National Bureau of Standards which, it is claimed, may have advantages for the separation of liquids which differ in their molecular weights or vapour pressures but have similar properties otherwise. The columns are arranged in such a manner that the condensate, moving by gravity, flows from column to column in one direction while all the residuals move in the opposite direction. This facilitates counter-current refluxing with automatic re-combination of fractions and the desired separation of fractions is accomplished in a single operation. Potential applications mentioned : Concentration of vitamins, vegetable oils, animal fats, lubricating oils and high-boiling petroleum fractions.

Sommaire : Une courte description d'un appareil à distillation moléculaire a reflux at à plusieurs " colonnes "

Note by Anon. *Chem. Engng. News*, **28**, 26 June 1950, 2178
See Also : *Chem. Engng.*, **57**, August 1950, 131

Fireproof Still

42/III

United States. This is a short note with photograph attached of a vacuum still protected by a safety device against fire and explosion. The still normally working under a vacuum of 29½ in. Hg is used in connection with a process where any leakage of air is dangerous. The device consists of a steel cylinder with a content of 5 lbs. of liquid carbon dioxide under high pressure. The cylinder is connected to the top of the still and is fitted with a discharge head operated electrically. The switch in the circuit is actuated, if the vacuum in the still drops to 25 in. Hg.

Sommaire : Dispositif de sécurité pour un appareil à distillation sous vide de matières inflammables.

Note by W. E. Morgan, Jr. *Chem. Engng.*, **57**, March 1950, 121

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

39 — MISCELLANEOUS PROCESSES —39

Improvements Relating to Enamelling — 43/III

United Kingdom. A method of producing vitreous enamel coatings on metal articles aiming at the improvement of their resistance to chemical attack. It is proposed to carry out drying of ground and cover coat under reduced pressure. The vacuum recommended is 4 mm.Hg. De-gassing of the metal is required prior to coating.

Sommaire : Procédé de revêtement d'articles en métal d'une couche d'émail " vitreux ".

A. W. Elmes, H. M. Glass, & Imperial Chemical Industries, *Brit. Pat.* 626, 008

De-Odorization of Vegetable Oils — 44/III

United Kingdom. De-Odorization is the final stage in the purification of oils and fats, prior to their incorporation into edible products. A plant is described which consists essentially of a de-odorizing vessel (capacity 15 tons), designed to operate under high vacuum, a vacuum-augmenter or thermo-compressor and a ' catch-all ' trap retaining the oil entrained in the vapour flow. As a rule a barometric jet condenser is employed. The residual air from the condenser is evacuated by a two-stage steam ejector. The effect of varying temperature and vacuum was investigated and it was found that the time needed for de-odorization and the blowing-steam requirements are in an inverse ratio to the vapour pressure of the volatile oil components at the operating temperature, and also to the vapour pressure of the principle fatty acids, *i.e.*, as the temperature is raised, the processing time is reduced. A table is attached, demonstrating this condition :—

Time Required for the Processing of Cottonseed Oil Products

Temperature °F	*Vapour Pressure of Oleic Acid mm. Hg.*	*Time Hrs.*
350	1.4	16.0
400	4.6	4.9
450	15.0	1.5
500	43.0	0.5

Sommaire : Description d'un appareil pour la dé-odorisation des huiles végétales.

Article by A. Arneil *Chem. & Industr.* 7-1-50. 6

Cream Fondant by Vacuum Methods — 45/III

United States. At a conference held at Bethlehem, Pa., much emphasis was given to the many advantages offered by vacuum processing candies, cream fondant and other confections. In this connection a new continuous vacuum method for making cream was mentioned. A cream with 84% sugar content and 16% glucose content was produced by cooking the syrup in jacketed kettles to 242°F. Subsequently it was drawn through a special nozzle into a vacuum chamber, where its temperature was reduced to approximately 110°C ready for beating.

Sommaire : Une méthode de fabrication de la crème fondante sous vide.

Note by Anon. *Food Industr.*, **22**, June 1950, 136

Vacuum Processing Apples Prevents Browning — 46/III

United States. A report is quoted on ' Syrup Impregnation of Apple Slices for Freezing ' which gives a comparison between two vacuum processes for inhibiting browning of apple slices in connection with freezing or canning. The first method provides for the submersion of the apple slices in a 30% sucrose solution containing 0.1% ascorbic and 0.1% citric acid and subjecting the apple slices in this condition to a vacuum of 26 in. for a period of 30 minutes. The second process provides for a solution of different composition, *i.e.*, 5%NaCl containing 1% citric acid. Both methods result in a brightly coloured, slightly translucent appearance of the apple slices. The texture of brine-treated slices was firmer than that of the syrup-treated samples.

Sommaire : Traitement des pommes sous vide pour empêcher la décoloration.

Note by Anon. *Food Industr.*, **22**, June 1950, 124

Chemistry in Food Preservation — 47/III

See Abstract No. : 15/I

Vacuum Cooling — 48/III

United States. The benefits of vacuum cooling foodstuff are discussed with particular reference to leafy vegetables or similar substances which require special measures for their preservation until processing is complete. Experiments with lettuce carried out by the Department of Agriculture are mentioned. It is stated that the process in question could be applied to bulk material and pre-packaged goods and may be commercialised in the current year.

Sommaire : Une courte note sur les avantages du refroidissement des matières alimentaires sous vide.

Note by Anon. *Food Industr.*, **22**, Feb. 1950, 9

Vol. 1 1951

IV — Special Subsidiary Subjects — IV

Abstract No. and References

40 — GASES AND VAPOURS — 40

Gas Analysis — 1/IV

United States. This is a survey of developments in 1949. Among others vacuum extraction equipment is discussed and also the application of combined vacuum fusion and ionic bombardments methods in connection with the investigation of the gas content of aluminium. It is stated however, that at least in its present state of development the ionic bombardment method is not fully satisfactory. In connection with volumetric procedures low-pressure micro-analysers are mentioned and a micro-doser for the introduction of minute amounts of gas into a vacuum system. 358 references.

Sommaire : Revue des progrès dans la technique du dosage des gaz au cours de l'année 1949.

Article by L. K. Nash *Analyt. Chem.*, **22**, Jan. 1950, 108-118

Detection and Measurement of Organic Vapours — 2/IV

United Kingdom. A discharge tube with Pt or Al wire electrodes placed inside tubes has two additional side tubes, one of which is connected to a vacuum pump and the other to a short tube, cupped at one end and having a plug of plaster of Paris in the bore, which acts as an air leak. Pieces of cotton wool moistened with the liquid to be tested are placed in the cup. Changes in the spectrum of the glow discharge due to the presence of the vapour of organic compounds are measured quantitatively by directing the light from the discharge tube onto a photo-electric cell. The amplified output from the cell actuates a milliammeter.

Sommaire : On utilise les changements dans le spectre d'une décharge pour indiquer la présence de matières organiques dans une enceinte à vide.

E. O. Powell and G. R. Smithson *Brit. Pat.* 628,824

Determination of Liquid-Vapour Equilibria — 3/IV

United States. A new form of a dew point bubble-point apparatus is described consisting essentially of a graduated cylinder tapered into a capillary, a large mercury reservoir and a glass tube used for balancing the mercury height in the graduated cylinder. The apparatus is suitable for use at sub-atmospheric pressures and temperatures up to 50°C but by replacing the vacuum pump with a pressure pump the range can be extended to several atmospheres. The apparatus is recommended for the determination of molecular weights of liquids and for the analysis of such binary liquid systems which it is difficult to analyse by refractive index or density. Finally it is recommended for the study of liquid-vapour equilibrium data of ternary or higher order systems.

Sommaire · Description d'un appareil pour la mesure des equilibres liquide-vapeurs.

Article by M. Feller and H. J. McDonald *Analyt. Chem.*, **22**, Feb. 1950, 338-340

A Portable Gas Blending System — 4/IV

United States. An article on the " Instrumentation and Auxiliary Equipment for Automatic Infra-Red Gas Analysers " mentions a gas blending system as an auxiliary to the analyser, developed for the purpose of checking the range and sensitivity of that instrument. A diagram of the gas blending system and a photograph are shown. The system is entirely made of glass. In operation the system is evacuated first and then a pure gas is admitted to the input reservoir. By means of a Toepler pump the gas sample is transferred to the mixing reservoir. The input reservoir is evacuated again and the next component gas admitted. When all component gases have been introduced and their partial pressures determined, the gases are thoroughly mixed by the action of a circulating pump of the sealed centrifugal type. Evacuation is carried out by a mechanical vacuum pump. The thorough mixing of the blend is essential to obtain homogeneity and reproducible calibration data. The Infra-red gas analyser described in this article is used for process control in petroleum production.

Sommaire : Un appareil portatif pour le mélange des gaz.

Article by W. J. Happel and N. D. Coggeshall *Instruments*, **23**, June 1950, 552-556

Glass Circulating Pump — 5/IV

See Abstract No. : 2/II

Measurement of Appearance Potentials of Ions Produced by Electron Impact, Using a Mass Spectrometer — 6/IV

United Kingdom. In measuring the appearance potentials of ions using a mass spectrometer two gases are admitted to the instrument simultaneously : A standard gas and the gas under investigation. Measurement of the ion beam intensities is made as a function of electron energy for both : The ion, the appearance potential of which is to be established, and the ion, the appearance potential of which is known. These measurements are made for a range of a few volts above the potentials on which the beams are first detected and several rather arbitrary methods exist for deducing the difference between the two potentials. In this communication a new method is described which has been employed in connection with work on dissociation processes in certain gases, using a Nier-type mass spectrometer. Details of the method are given and tabulated results are added, obtained for various ions with argon as a standard gas.

Sommaire : Méthode de mesure de la tension requise pour que les ions produits par bombardement électronique soient enregistrés par le spectromètre de masse.

Letter by J. Warren *Nature*, **164**, 20.5.50, 810-811

IV — Special Subsidiary Subjects — IV

Contd.

Abstract No. and References

Method of Handling and Storing Liqufied Gases 7/IV

United Kingdom. Details are given of equipment installed at the Clarendon Laboratory, Oxford, England, for storing and conveying liquid oxygen nitrogen and similar substances. Liquid oxygen is stored in a vessel of 400 l. capacity. It consists of a cylindrical container made from welded copper and is surrounded by radiation shields. This cylinder is suspended in a housing provided by a converted boiler. A rough vacuum of a few mm.Hg is maintained in the boiler and a high vacuum of the order of 2×10^{-6} mm.Hg in the interior of the cylinder. The high vacuum is produced by a continuously running oil diffusion pump discharging into a 120 l. reservoir. A mechanical pump connected to the system comes into action as soon as the backing pressure of the diffusion pump rises to about 0.1 mm.Hg. The rough vacuum in the boiler is maintained by an additional mechanical pump, operating intermittently. An oil manometer indicating the difference in pressure across the outer copper vessel acts as a safety valve at the same time. In addition, vacuum syphons, a liquid air converter and various control instruments are described.

Sommaire : Description des installations aux laboratoires Clarendon, à Oxford, pour la préservation des gaz liquefiés.

Article by A. J. Croft and D. O. Jones *Brit. J. Appl. Phys.* 1, June 1950 137-143

On Certain Phenomena Concerning the Release of Gases Observed in Connection with the Production of Low-Voltage Incandescent Lamps 8/IV

See Abstract No. : 6/I

41 — FREEZE DRYING — 41

Fractionation of Blood Plasma

United States. A description of a plant erected in 1943 and capable of handling plasma obtained from approximately 8,000 donors a week. 9/IV

The primary product of the plant is albumin and gamma-globulins. To ensure cleanliness, the whole of the building is maintained under positive air pressure and the processing equipment is so constructed that the plasma and all the reagents are allowed to come in contact only with glass, rubber or stainless steel. Elaborate precautions have been taken to keep all reagents pyrogen-free. The actual fractionation of the plasma is carried out by a process of continuous centrifugation, several units being used instead of one to be able to process small amounts of blood without appreciable loss. Conveying of the material is carried out by air under pressure but in some instances suction proved to be more convenient.

The removal of alcohol from the protein precipitates is effected by drying from the frozen state. The paste is suspended in distilled water reducing the alcohol concentration to 20% or less. Stainless steel containers holding 1½ gal. of this suspension are rotated in a freezing bath after chilling at a temperature of -70°. This results in the formation of a frozen shell of material inside the container. Subsequently, the container is transferred to a high vacuum plant and sublimation allowed to proceed. The vacuum required is produced by steam ejectors, the operational pressure is between 200-300 μ. Details are given of the plant and important plant components as for instance safety valves.

Sommaire : Description d'un appareil pour la distillatien du plasma sanguin sur une grande échelle. On inclut aussi un exposé sur l'élimination de l'alcool par sublimation sous vide à basse température.

Article by J. B. Lesh, K. Schultz and J. D. Porsche *Industr. Engng. Chem.*, 42 July 1950 1376-1380

New Techniques in Auto-Radiography 10/IV

United Kingdom. The preparation of tissues for auto-radiography presents two problems. (1) The danger of losing the soluble tracer and (2) the enormous radiation intensities needed for a successful exposure. Details are given of a procedure which largely overcomes these difficulties. The method reported uses a freeze drying technique similar to that developed by Gersh. Equipment for drying in preparing such specimens consists essentially of a drying chamber connected to a diffusion pump with a cooling jacket connected to the rotary pump. The jacket is cooled by evaporation of liquid methylchloride under reduced pressure. Before the frozen specimen is introduced into the chamber paraffin wax is poured into the bottom of the drying chamber and, after degassing, is allowed to solidify. After the introduction of the specimen the chamber is evacuated and when the specimen is dry it is infiltrated with wax in situ. After embedding, the block is sectioned and the ribbon is floated on a mercury surface. A suitable photographic emulsion, *e.g.*, Kodak NT4, is then placed onto the floated section and exposed to the tracer action. Sections giving a Geiger—Muller β particle count of only 10 counts/min./sq.mm. give autoradiographs of excellent contrast after an exposure of a few hours.

Sommaire : Méthode de préparation des tissues pour l'auto-radiographie.

Article by J. E. Harris, J. F. Sloane and T. P. King *Nature*, 166, 1.7.1950 25-26

Preservation of Fungal Sporophores 11/IV

United Kingdom. It has been observed that fleshy sporophores of the higher fungi are not affected by frost but collapse when thawn out. Where animal tissues are to be preserved by means of drying in the frozen state

Vol. 1 1951

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

it is important, therefore, to overcome this drawback. A method is suggested which will facilitate preservation of the morphological character of such tissues by means of immersing the sporophores for a short period in melted wax following drying in the frozen state and after their removal from the desiccator. The actual material used is " Seekay " artificial wax grade R93. The immersion period should not be longer than 30 sec. otherwise shrinkage will be caused. Surplus wax can be removed by the application of a suitable solvent and in this manner the final degree of gloss on the specimen can be controlled. The " Seekay " wax has been found to be superior to paraffin wax in this application. It has greater hardness and strength, is rapidly penetrating, highly water-resistant and non-inflammable.

Sommaire : On decrit une méthode pour la preservation des grains fongueux tout en empêchant leur destruction durant le dégel.

Letter by W. Nielson Jones and J. W. Trevan *Nature*, 165, 29.4.50 692

Technical Developments in Fermentation Processes 12/IV

United Kingdom. This is a survey of up-to-date fermentation techniques. It is written from the angle of a bio-chemist rather than that of a chemical engineer. Under the heading of "Biological Aspects", it is pointed out that the early stages of developing a fermentation process are usually very troublesome due to the capriciousness of the micro-organisms dealt with in most cases. The technique of sub-culture has to be worked out separately for each organism and whilst one type may need recourse to natural media at frequent intervals, others require constant " Plating out on Petri-dishes " The author emphasises that the technique of freeze drying of suitable cultures which has recently been applied on a large scale has proved to be of great value in this connection, ensuring stability of the organisms, but care must be taken not to interpose too many " generations " between the master culture, whether freeze-dried or not, and the inoculum which is finally introduced into the fermentation vessel.

Sommaire : L'application de la dessication sous vide aux problèmes de la fermentation.

Article by H. S. Corran *Chem. & Industr.* 4.2.50 86-88

Chemistry in Food Preservation 13/IV

See Abstract No. : 15/I

45 — ELECTRONICS — 45

Vacuum Factor of the Oxide Cathode Valve 14/IV

United Kingdom. It is generally believed that the life of a high vacuum oxide cathode valve depends on the quantity of residual gas left in the valve after processing. Measurement of such residual gas content can be effected by ascertaining the number of positive ions formed in an electron flow of determined quantity. In the experiments described here the ions formed in such an electron stream were collected on a negative electrode introduced into the tube and the vacuum factor expressed in $\mu\mu$ A/mA electron current. Investigations were carried out on vacuum factor/time characteristics, variation of ionization probability with electron energy and the influence of the oxide cathode temperature, or that of the envelope on the vacuum factor. It was found that the value of the vacuum factor is a direct function of anode voltage, independent of anode current, anode temperature, oxide cathode temperature and finally independent of the envelope temperature, providing gas evolution is prohibited. It appears that the vacuum factor is solely dependent on the energy of the electrons striking the anode. It is suggested that these findings have a bearing on the interpretation of ionization gauge measurements at pressures below 1×10^{-6} mm.Hg.

Sommaire : Etude des facteurs déterminants la durée des tubes à cathode à oxyde.

Article by G. H. Metson *Brit. J. Appl. Phys.*, 1, March 1950, 73-77

Thermionic Emission of Thin Films of Alkaline Earth Deposited by Evaporation 15/IV

United States. The paper deals with mono-molecular films BaO or SrO deposited by evaporation on clean tungsten or molybdenum surfaces after precautions had been taken to eliminate effects caused by excess metal of the oxide or by heating. Evidence is given that films of pure alkaline oxide with a thickness of 1-25 molecular layers deposited by evaporation can lower the thermionic work function of clean receiver metal by as much as 3.5 volts. An explanation is given for this effect. Details of the experimental procedures involved are given and comparisons made with conditions prevailing in the case of bulk oxide coatings. There is a similarity in the magnitude of emission between both systems, but this is not to be taken as evidence for a similarity in the nature of the emission process. It is stated that the emission from both the thick-layer practical cathode and thin films of the type described have increased by a factor of more than 1,000 in the course of the last 30 years, but it is also stated that further work is required before a decision can be made whether the thin film mechanism is of any importance to practical cathodes.

Sommaire : Etude sur l'émission thermionique des couches minces d'oxyde déposées par évaporation sous vide.

Article by G. E. Moore and H. W. Allison *Phys. Rev.*, 77, 15.1.50 246-257

Design Study of a 3-Bev Proton Accelerator 16/IV

United States. This article describes the essential design features of a proton accelerator at present being built. The purpose of the plant is to accelerate protons injected from a 4,000,000-volt Van der Graaff machine to

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

energies of the order of 3 billion-volt. The protons entering the accelerator are deflected electrostatically into a race-track orbit which consists of 4 quadrants of 30 ft. radius of curvatures and 4 straight sections of 10 ft. each.

Details for the pumping system are given. The maximum permissible pressure is 1×10^{-5} mm.Hg. There are 12 pumping stations located equidistant around the periphery of the chamber. A 28 in. oil diffusion pump is allocated to each station. The rough backing vacuum for all diffusion pumps is catered for by 4 centrally located fore-pumps. The speed of each pumping station is 2,500 l. per sec.

Sommaire : On donne des détails sur un accélérateur de protons qui vient d'être construit aux États-Unis, anisi qu'une description complète du système de pompage.

Article by M. S. Livingston J. P. Blewett G. K. Green and L. H. Haworth *Rev. Sci. Instrum.*, 21. Jan., 50 5-24

Automatic Regulation of Thermionic Emission

United Kingdom. A circuit is described which automatically controls the emission of thermionic devices for large changes of emission characteristics, and is also independent of large mains voltage fluctuations. In this circuit the power required to heat the emissive surface is controlled by two valves which have the two primary windings of the filament transformer as their load. Part of the electron current to be stabilised is used to control the two valves *via* a pre-amplifier, so that an increase in emission will reduce the current passed by the valves, and thus the filament current. A phase-shift network is used to obtain high stabilization ratios without setting up oscillations in the feed-back network when controlling massive filaments. A circuit is given applying these principles to the emission control of an ionization gauge in which the grid current is kept constant to within 0.5% under all conditions.

Sommaire : Description d'un circuit permettant le contrôle automatique de l'émission d'éléments thermo-ioniques qui pourraient avoir de grands changement dans leur émissivité.

17/IV

Article by W. Steckelmacher and S. Van der Meer, *J. Sci. Instrum.*, 27, July 1950, 189-191

Manufacturing Metallised Picture Tubes

See Abstract No. : 7/III

18/IV

Electrolytic Polishing and Vacuum Engineering

See Abstract No. : 4/I

19/IV

Helium Disappearance in Arc Discharge Tubes

20/IV

United States. This is a short report on methodic experiments into the nature of " clean-up " phenomena conducted by Martin Reddan and published by the U.S. National Bureau of Standards. A tantalum probe wire was placed in a specially designed discharge tube to collect the positive ions. The tube was filled with helium and was fitted in addition with an oxide-coated cathode and a nickel anode. During operation the rate of gas clean-up was measured for various negative d.c. potentials on the probe in the presence of an arc-discharge. The results of this experiment are shown in a graph and are intended to assist tube designers in choosing the right structural materials to ensure long life of the tube.

Sommaire : Une étude sur le phénomène d'absorption qui a lieu dans les tubes à décharge dans la vapeur d'hélium.

Note by M. Lorant *Electronic Engng.*, 22, March 1950, 108-109

47 — GLASS PROCESSING — 47

Device for Making a Vacuum-Tight Connection Between Two Metallic Parts of Different Electric Potential

21/IV

United Kingdom. The method described is claimed to avoid all dangerous tensile stress in the glass and metallic elements which form part of the connection. The sleeves of the two metallic components to be joined are shaped in a manner which will facilitate the application of glass flux as a joining material. The sleeve of the outer component has a crimped edge and the sleeve of the inner component a groove.

Sommaire : Description d'un type spécial de joint verre-métal.

Brown, Boveri & Co., *Brit. Pat.* 635, 444

A Method of Making Glass-To-Metal Seals

22/IV

United States. A method is described of producing such seals by an electro-forming process. The joining of a Pyrex glass and copper tube is carried out in the following manner :—The glass tube is coated at one end with a ceramic-type silver paint which is subsequently fired on the copper tube and produces a strong glass-silver bond. A wax form is inserted and its surface made electrically conducting by the application of air-drying silver paint. This facilitates the deposition of a layer of copper (by electro-plating) over the entire surface of the joint. Finally, the form is removed by melting out the wax. Seals of this type have been found to be vacuum tight.

Sommaire : On décrit une méthode galvano-plastique de fabrication des joints verre-métal.

Note by N. A. Eckstein J. W. Fitzgerald and C. A. Boyd *Rev. Sci. Instrum.*, 21, April 1950, 398-399

Vol. 1 1951

Cumulative Author Index

Author	Title of Publication	Abstr. No.
Alexander, P.	Improvements i.o.r.t. Methods of Eliminating Moisture from the Surfaces of Moisture Absorbant Sheet Material on Which Metal is to be Deposited By Thermal Evaporation in a Vacuum.	24/III
Allison, H. W., et al.	Thermionic Emission of Thin Films of Alkaline Earth Deposited by Evaporation.	15/IV
Andrade, E. N. da C., et al.	The Thermal Etching of Single Crystals of Cadmium.	12/I
Anon.	The Electronic Telescope.	26/II
Anon.	Porcelain, Its Manufacture and Uses.	33/II
Anon.	Gilding the Lily.	10/III
Anon.	Seeing Molecules.	11/III
Anon.	Uranium in Microscopy.	12/III
Anon.	Cathodic Vacuum Etching Process.	23/III
Anon.	Distillation Process for Fats.	39/III
Anon. (R.L.K.)	Defatting Distillation.	40/III
Anon.	Multi-Column Reflux Still.	41/III
Anon.	Cream Fondant by Vacuum Methods.	45/III
Anon.	Vacuum Processing Apples Prevents Browning.	46/III
Anon.	Vacuum Cooling.	48/III
Arneil, A., et al.	De-Odorization of Vegetable Oils.	44/III
Atkins, W. F., et al.	The Design of a Large Vacuum Valve for Application to an Electric Furnace.	35/II
Axelbank, M.	A Rotary McLeod Gauge.	7/II
Backus, R. C., et al.	The Use of Spraying Methods and of Volatile Suspending Media in the Preparation of Specimens for Electron Microscopy.	20/II
Beach, A. L., et al.	A Vacuum Fusion Furnace for the Analysis of Gases in Metals.	31/III
Birks, L. S., et al.	Particle Size of Evaporated Gold.	15/III
Blasco, E., et al.	A New Pirani-Type Vacuum Gauge.	10/II
Blewett, J. P., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Bockris, J. O'M., et al.	A Vacuum Furnace for Use in the Temperature Range of 1000°-2000°C.	32/III
Boyd, C. A., et al.	A Method of Making Glass-To-Metal Seals.	22/IV
British-Thomson-Houston Co. Ltd.	Improvements i.o.r.t. Non-Reflecting Optical Bodies.	3/III
Brown, J.	Modern Practice in Investment Casting.	26/III
Brown Boveri & Co., A. G.	Device for Making a Vacuum-Tight Connection Between Two Metallic Parts of Different Electric Potential.	21/IV
Burmaster, K. E.	Experimental High Megohm Resistors.	8/I
Butler, C. P., et al.	A Motor for Use in Vacuum Systems.	37/II
Carpenter, F. E., et al.	A Motor for Use in Vacuum Systems.	37/II
Clark, J. W., et al.	An Automatic Control of High Vacuum Systems.	39/II
Clark, W. J.	Tele-recording of Thickness, Pressure Flow & Other Physical Quantities Using a Simple Electromagnetic Circuit.	9/II
Coggeshall, N. D., et al.	A Portable Gas Blending System.	4/IV
Corran, H. S.	Technical Developments of Fermentation Processes.	12/IV
Coslett, V. E.	Preparation of Specimens for the Electron Microscope.	8/III
Coulson, J. M., et al.	Manufacture of Mononitrotoluene.	37/III
Courtney-Pratt, J. S.	Direct Optical Measurements of the Length of Organic Molecules.	14/I
Croft, A. J., et al.	Method of Handling & Storing Liquefied Gases.	7/IV
Derge, G.	Analysis of Oxygen in Titanium.	30/III
Deutsch, Z. G., et al.	Mass Spectrometer for Leak Detection.	41/II
Duckworth, J. E.	A Large Dempster Type Double-Focusing Mass Spectrograph.	27/II
Dunoyer, L.	On Certain Phenomena Concerning the Release of Gases Observed in the Production of Low-Voltage Incandescent Lamps.	6/I
Eckstein, N. A., et al.	A Method of Making Glass-To-Metal Seals.	22/IV
W. Edwards & Co. (London) Ltd., et al.	Improvements in Measuring Devices Dependent on Radiometer Action.	6/II
Elmes, A. W., et al.	Improvements Relating to Enamelling.	43/III
Ewald, E. R.	Manufacturing Metallised Picture Tubes.	7/III

ol.
1
951

Cumulative Author Index

Contd.

Author	Title of Publication	Abstr. No.
Fast, J. D.	Apparatus for the Preparation of Metals with an Exactly Known Content of Impurities.	33/III
Fein, M. L., et al.	An Improved Manostat and Manometer.	40/II
Feller, M., et al.	Determination of Liquid-Vapour Equilibria.	3/IV
Fitzgerald, J. W., et al.	A Method of Making Glass-To-Metal Seals.	22/IV
Fortune, W. B., et al.	Antibiotics Development.	25/III
Garfitt, R. G., et al.	A Three-Stage Electron Microscope with Stereographic Dark Field, & Electron Diffraction Capabilities.	19/II
Garrod, R. I.	A Low-Impedence High Vacuum Valve.	36/II
Gibson, A. F., et al.	The Photo-Conductivity of Bismuth Sulphide and Bismuth Telluride.	16/III
Glass, H. M., et al.	Improvements Relating to Enamelling.	43/III
Glober, J. A., et al.	Glass Circulating Pump.	2/II
Green, G. K., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Groszkowski, J.	A McLeod Gauge of Multiple Compression.	8/II
Guldner, W. G.	Determination of Carbon in Ferrous Alloys.	29/III
Guldner, W. G., et al.	A Vacuum Fusion Furnace for the Analysis of Gases in Metals.	31/III
Haine, M. E., et al.	A Three-Stage Electron Microscope with Stereographic Dark Field, and Electron Diffraction Capabilities.	19/II
Hall, C. E.	A Low Temperature Replica Method for Electron Microscopy.	9/III
Hamm, F. A.	Electron Microscope.	17/II
Hamm, F. A.	Symposium on Thin Films.	2/III
Happel, W. J., et al.	A Portable Gas Blending System.	4/IV
Harris, J. E., et al.	New Techniques in Autoradiography.	10/IV
Haworth, L. H., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Heller, R. B.	Cutting Flat Vacuum Gaskets on a Lathe.	38/II
Helwig, G., et al.	The Structure of Obliquely Evaporated Films & Their Influence on the Formation of Sub-Microscopic Surface Irregularities.	13/III
Hickman, K. C. D.	High Vacuum Distillation.	36/III
Hicks, T. J.	How To Figure Reciprocating Vacuum Pumps.	1/II
Hipple, J. A., et al.	Mass Spectrometry.	16/II
Ilfeld, R. M.	A Recording Mass Spectrometer.	28/II
Imperial Chemical Industries, et al.	Improvements Relating to Enamelling.	43/III
Jenkins, G. C. H., et al.	The Design of a Large Vacuum Valve for Application to an Electric Furnace.	35/II
Johnson, F. C., et al.	Glass Circulating Pump.	2/II
Johnson, G. W.	Surface Tension Effects on Oriented Thin Silver Films.	14/III
Johnson, P. D.	Behaviour of Refractory Oxides of Metals Alone & in Combination in Vacuo at High Temperatures.	34/II
Jolly, W. P., et al.	The Pirani Effect in a Thermionic Filament as a Means of Measuring Low Pressures.	4/II
Jones, D. O., et al.	Method of Handling & Storing Liquefied Gases.	7/IV
Keeping, Jr., G. R.	On the Growth of Metallic Crystals from the Vapour Phase.	5/III
Kern, R. A., et al.	The Apparent Size of Objects as Observed in the Electron Microscope.	32/II
Kern, S. F., et al.	The Apparent Size of Objects as Observed in the Electron Microscope.	32/II
King, T. P., et al.	New Techniques in Autoradiography.	10/IV
Koenig, H., et al.	The Structure of Obliquely Evaporated Films & Their Influence on the Formation of Sub-Microscopic Surface Irregularities.	13/III
Kroll, W. J.	Improvements in Processes & Apparatus for the Treatment of Hafnium, Zirconium, Titanium, Thorium and Their Alloys, in the Molten State.	27/III
Kroll, W. J., et al.	Production of Malleable Zirconium.	35/III
Lampitt, L. H.	Chemistry in Food Preservation.	15/I
Leduc, P.	Precautions to be Taken in the Use of the Philips Gauge.	15/II
Lesh, J. B., et al.	Fractionation of Blood Plasma.	9/IV
Livingston, M. S., et al.	Design Study for a Three-Bev Proton Accelerator.	16/IV
Lloyd, J. T.	An Audible Vacuum Leak Detector.	42/II

Vol. 1 1951

Cumulative Author Index

Contd.

Author	Title of Publication	Abstr. No.
Longini, R. L.	Evaporation & Outgassing in an Inert Atmosphere.	11/I
Lorant, M.	Helium Disappearance in Arc Discharge Tubes.	20/IV
Matalon, R., et al.	A Simplified Design for a Molecular Still of the Horizontal Type.	38/III
McCormick, S. L., et al.	Antibiotics Development.	25/III
McCutcheon, D. M., et al.	Cathodic Vacuum Etching of Metals.	23/III
McDonald, H. J., et al.	Determination of Liquid-Vapour Equilibria.	3/IV
McFee, R. H.	A Method of Supplying Low-Pressure Gases to a Vacuum System.	5/I
Meer, S. van der, et al.	Automatic Regulation of Thermionic Emission.	17/IV
Metson, G. H.,	Vacuum Factor of the Oxide Cathode Valve.	14/IV
Miranda, L., et al.	A New Pirani-Type Vacuum Gauge.	10/II
Moore, G. E., et al.	Thermionic Emission of Thin Films of Alkaline Earth Deposited by Evaporation.	15/IV
Morgan, Jr. W. E.	Fireproof Still.	42/III
Moss, T. S., et al.	The Photo-Conductivity of Bismuth Sulphide and Bismuth Telluride.	16/III
Nash, L. K.	Gas Analysis.	1/IV
Nielson Jones, W., et al.	Preservation of Fungal Sporophores.	11/IV
Nienhuis, K., et al.	Construction & Applications of a New Design of the Philips-Type Vacuum Gauge.	11/II
Nineuil, P.	Electrolytic Polishing & Vacuum Engineering	4/I
Page, R. S., et al.	A Three-Stage Electron Microscope with Stereographic Dark Field, & Electron Diffraction Capabilities.	19/II
Pahl, W., et al.	Cathodic Vacuum Etching of Metals.	23/III
Penning, F. M., et al.	Construction & Applications of a New Design of the Philips-Type Vacuum Gauge.	11/II
Porsche, J. D., et al.	Fractionation of Blood Plasma.	9/IV
Powell, E. O., et al.	Detection & Measurement of Organic Vapours.	2/IV
Raible, F., et al.	Mass Spectrometer for Leak Detection.	41/II
Randall, R. F. Y., et al.	The Thermal Etching of Single Crystals of Cadmium.	12/I
Ratchford, W. P., et al.	An Improved Manostat and Manometer.	40/II
Rhode-Hamel, H. W., et al.	Antibiotics Development.	25/III
St. Clair, H. W., et al.	Low-Pressure Distillation of Zinc, from Al-Zn Alloy.	34/III
Schneider, M.	Metallising by High Vacuum.	1/III
Schultz, K., et al.	Fractionation of Blood Plasma.	9/IV
Shepherd, M., et al.	Mass Spectrometry.	16/II
Sloane, J. F., et al.	New Techniques in Autoradiography.	10/IV
Smith, C. C., et al.	A Simplified Design for a Molecular Still of the Horizontal Type.	38/III
Smithson, G. R., et al	Detection & Measurement of Organic Vapours.	2/IV
Snyder, F. L.	Insulation for High Voltage Apparatus.	7/I
Spendlove, M. J., et al.	Low-Pressure Distillation of Zinc, from Al-Zn Alloy.	34/III
Spiers, L., et al.	The Pirani Effect in a Thermionic Filament as a Means of Measuring Low Pressures.	4/II
Steckelmacher, W., et al.	Improvements in Measuring Devices Dependent on Radiometer Action.	6/II
Steckelmacher, W., et al.	Automatic Regulation of Thermionic Emission.	17/IV
Stefaniak, J. J., et al.	Antibiotics Development.	25/III
Stephens, W. W., et al.	Production of Malleable Zirconium.	35/III
Ternisien, J.	Electron Diffraction.	18/II
Tomlinson, J. W., et al.	A Vacuum Furnace for Use in the Temperature Range of 1000°-2000°C.	32/III
Trevan, J. W., et al.	Preservation of Fungal Sporophores.	11/IV
Warner, F. E., et al.	Manufacture of Mononitrotoluene.	37/III
Warren, J.	Measurement of Appearance Portentials of Ions Produced by Electron Impact, Using a Mass Spectrometer.	6/IV
Wilkinson, P. G., et al.	Particle Size of Evaporated Gold.	15/III
Williams, R. C., et al.	The Use of Spraying Methods & of Volatile Suspending Media in the Preparation of Specimens for Electron Microscopy.	20/II
Witts, G. H., et al.	An Automatic Control of High Vacuum Systems.	39/II

Abbreviations and Publishers of Journals Quoted in this Volume

Analyt. Chem.	Analytical Chemistry. American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Brit. J. Appl. Phys.	British Journal of Applied Physics. Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330 W. 42nd Street, New York, 18, N.Y., U.S.A.
Chem. Engng. News	Chemical Engineering News. American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Chem. Industr.	Chemical Industries. McGraw-Hill Publishing Co., Inc., 330 W. 42nd Street, New York, 18, N.Y., U.S.A.
Chem. and Drugg. Export Rev.	Chemist & Druggist Export Review, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Chem. and Industr.	Chemistry & Industry. Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Discovery	Discovery. Jarrold & Sons, Ltd., London Street, Norwich, England.
Electronic Engng.	Electronic Engineering. Morgan Brothers (Publishers), Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Electronics	Electronics. McGraw-Hill Publishing Co. Inc., 330 W. 42nd Street, New York 18, N.Y., U.S.A.
Food Industr.	Food Industries. McGraw-Hill Publishing Co., Inc., 330 W. 42nd Street, New York 18, N.Y., U.S.A.
Foundry	Foundry. Penton Publishing Co., Penton Bldg., Cleveland 13, Ohio, U.S.A.
Industr. Engng. Chem.	Industrial & Engineering Chemistry. The American Chemical Society, 20th & Northampton Streets, Easton, Pa., U.S.A.
Instrumentation	Instrumentation. Minneapolis-Honeywell Regulator Co. (Industrial Division) Wayne & Windrim Avenues, Philadelphia 44, Pa., U.S.A.
Instruments	Instruments. Instrument Publishing Co. Inc., 1117, Wolfendale Street, Pittsburgh 12, Pa., U.S.A.
J. Amer. Ceramic Soc.	Journal of the American Ceramic Soc. American Ceramic Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
J. Appl. Phys.	Journal of Applied Physics. American Institute of Physics, Prince & Lemon Streets, Lancaster, Pa., U.S.A.
J. Metals	Journal of Metals. American Institute of Mining and Metallurgical Engineers Inc., 29 W. 39th Street, New York 18, N.Y., U.S.A.
J. Sci. Instrum.	Journal of Scientific Instruments. Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Machinery	Machinery. Machinery Publishing Co. Ltd., National House, West Street, Brighton 1, England.
Metal Progr.	Metal Progress. American Society of Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Microtecnic	Microtecnic Scriptar S.A., 23, Avenue de la Gare, Lausanne, Switzerland.
Mod. Plastics	Modern Plastics. Modern Plastics Inc., 122 E. 42nd Street, New York 17, N.Y., U.S.A.
Nature (Lond.)	Nature. Macmillan & Co., St. Martin's Street, London, W.C.2, England.
Nucleonics	Nucleonics. McGraw-Hill Publishing Co. Inc., 330 W. 42nd Street, New York 18, N.Y., U.S.A.
Optik	Optik. Wissenschaftliche Verlagsgesellschaft m.b.H., Stuttgart, Germany.
Philips Techn. Rev.	Philips Technical Review Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, England.
Phys. Rev.	Physical Review. American Physical Society, Prince and Lemon Streets, Lancaster, Pa., U.S.A.

Vol. 1 1951

Abbreviations and Publishers of Journals Quoted in this Volume

Contd.

Proc. Phys. Soc.	Proceedings of the Physical Society. The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
The Times (Industr. Suppl.)	" The Times " Industrial Supplement. " The Times " Publishing Co. Ltd., Printing House Square, London, E.C.4, England.
The Times (Science Suppl.)	" The Times " Science Supplement. " The Times " Publishing Co. Ltd., Printing House Square, London, E.C.4, England.
Trans. Soc. Instrum. Technol.	Transactions of the Society of Instrument Technology. Society of Instrument Technology, 80, Bishopsgate, London, E.C.3, England.
Le Vide	Le Vide. Societe Francaise des Ingenieurs Techniciens du Vide, 44, Rue de Rennes, Paris (VIe), France.
Westinghouse Engr.	Westinghouse Engineer. Westinghouse Electric Corporation, 306, Fourth Avenue, P.O. Box 1017, Pittsburgh 30, Pa., U.S.A.